

000016416

**DELIVERABLE (COMBINED)  
231A, 231B, 231C, 231E**

**PONDSLUDGE TREATABILITY STUDY WORK PLAN,  
PONDCRETE TREATABILITY STUDY WORK PLAN,  
SALTCRETE TREATABILITY STUDY WORK PLAN AND  
CLARIFIER SLUDGE TREATABILITY STUDY WORK PLAN**

**FOR**

**EG&G ROCKY FLATS**


**PREPARED BY**

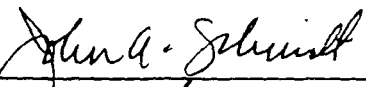
**HALLIBURTON NUS ENVIRONMENTAL CORPORATION  
PITTSBURGH, PENNSYLVANIA 15220**

**REVISION 1**

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## 1.0 PROJECT DESCRIPTION

This work plan describes the requirements and procedures for conducting a treatability study to develop chemical stabilization and solidification (CSS) formulations for several waste sources at the Rocky Flats Plant. This work is being conducted in support of the Solar Ponds/Pondcrete/Saltcrete Waste Processing project being conducted by HALLIBURTON NUS Environmental Corporation. The waste sources of concern, and that are to be included in the treatability study, are as follows:

- Solar Pond 207A sludge
- Solar Pond 207B (north, center, and south) sludge
- Solar Pond 207C sludge and water
- Clarifier sludge and water
- Pondcrete triwalls
- Pondcrete triwalls in metal containers
- Saltcrete triwalls
- Saltcrete triwalls in metal containers
- Saltcrete in half-crates

The following sections contain the site history, project purpose and description, and scope of work for the treatability study.

### 1.1 Site History and Description

During construction of the Rocky Flats Plant, a clay-lined solar evaporation pond was installed. The pond was designed for the impoundment of aqueous waste products discharged from the Process Waste Treatment Plant. The waste contained high levels of chemical contaminants, such as fluoride, nitrates, and various metallic ions. As a result of changing plant operations and environmental requirements, additional evaporation ponds were constructed. On

occasion these ponds were used for the disposal of untreated waste products, such as metallic lithium, acids, sewage sludge, plating residues, and several other wastes associated with operations at the Rocky Flats Plant.

Five solar evaporation ponds, designated ponds 207A, 207B (north, center, and south), and 207C, are located in the northeast corner of the plant as shown in Figure 1-1. The solar ponds were constructed to store and evaporate much of the process wastewater generated at the plant. The five ponds are currently being closed under RCRA.

Pondcrete was generated during solidification of pond sludge from Solar Pond 207A. Saltcrete was produced by the solidification of evaporator bottoms generated during routine wastewater treatment operations.

The clarifier contains approximately 25,000 gallons of water and sludge. This material originated from Pond 207A during the original pondcrete solidification project.

The wastes contained in the ponds, clarifier, pondcrete and saltcrete, are classified as hazardous mixed waste. EPA Hazardous Waste Numbers associated with the pond wastes, clarifier sludge, and pondcrete are F001, F002, F003, F005, F006, F007, F009, and D006. EPA Hazardous Waste Numbers associated with the saltcrete are F001, F002, F003, F005, F006, F007, and F009.

Waste characterization studies were used to determine the chemical composition of all of the above waste forms.

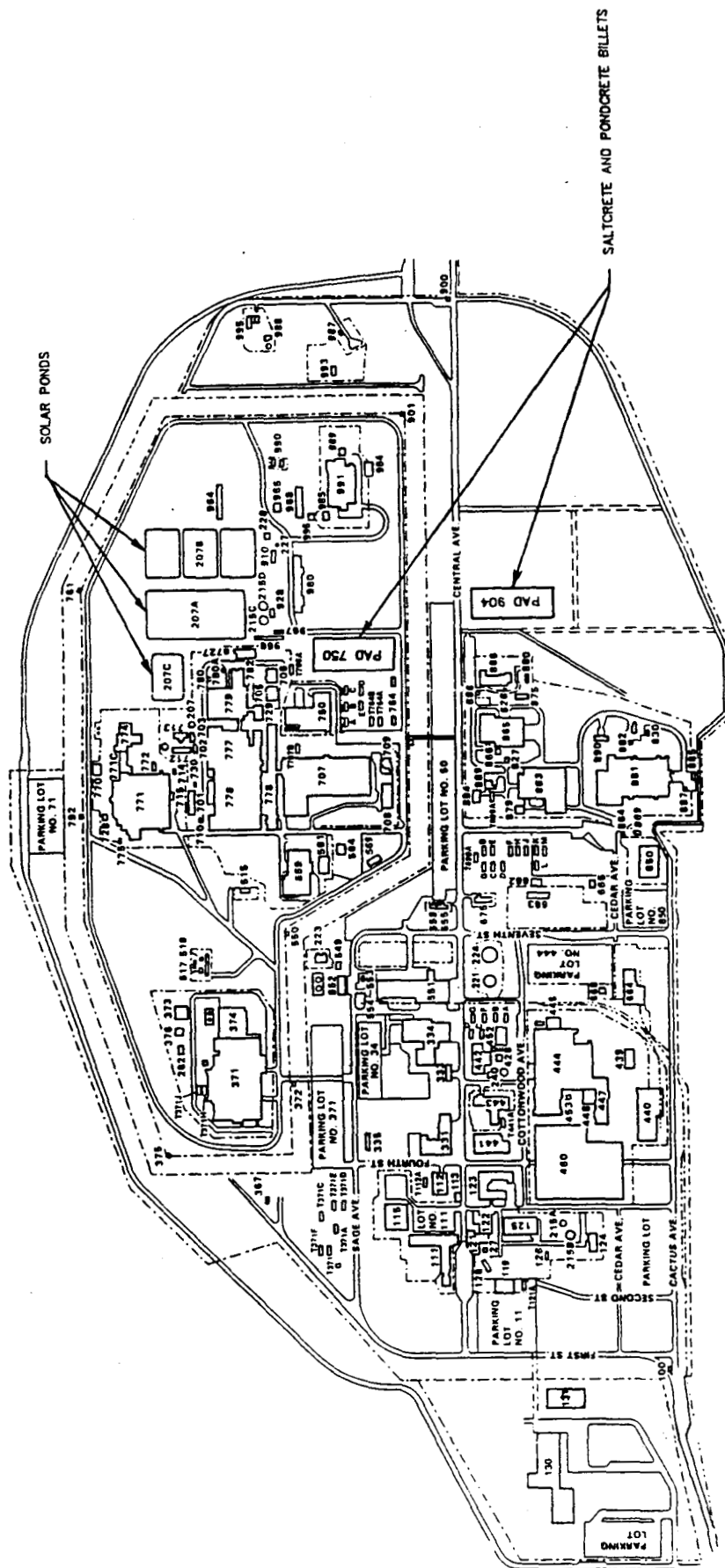


FIGURE 1-1



ROCKY FLATS COMPLEX  
ROCKY FLATS, GOLDEN, COLORADO  
NOT TO SCALE

### **1.1.1 Pond 207A**

Pond 207A covers approximately 2.7 acres. Studies are underway to define the volume of, and to characterize, the wastes contained in the pond. The pond contents have been classified as hazardous mixed waste. Constituents of concern based on previous analyses performed by Weston include cadmium, chromium, lead, mercury, and nickel. These metals may be present at levels that could cause EPA Land Disposal Restriction (LDR) treatment standards to be exceeded. Pond 207A sludge also contain percent levels of aluminum (3 to 4 percent), calcium (7 to 8 percent), iron (1 to 2 percent), potassium (1 to 2 percent), magnesium (1 to 2 percent), and sodium (2 to 3 percent). The pond sludge contains approximately 11 percent solids. No organic compounds were found above the detection limit in previous Weston analyses.

### **1.1.2 Ponds 207B North, Central, and South**

Each of these ponds covers approximately 0.98 acres. Studies are underway to define the volume of, and to characterize, the wastes contained in the ponds. Pond 207B-North also contains contaminated groundwater from the nearby, underlying french drain collection system. Constituents of concern based on previous analyses performed by Weston include cadmium, chromium, lead, mercury, and silver. These metals may be present at levels that could cause EPA Land Disposal Restriction (LDR) treatment standards to be exceeded. Pond sludges also contain percent levels of calcium (7 to 26 percent), chloride (<1 to 2 percent), magnesium (<1 to 2 percent), nitrate (<1 to 2 percent), and sodium (2 to 3 percent). The solids content of the sludges in these ponds range from less than 10 percent to more that 20 percent. Low concentrations (less than 1 ppm) of organics were detected during previous Weston analyses.

### **1.1.3 Pond 207C**

Pond 207C covers approximately 0.87 acres. Studies are underway to define the volume of, and to characterize, the wastes contained in the pond. The contents of the pond are classified as hazardous mixed waste and contain saturated salt concentrations. Constituents of concern based on previous analyses performed by Weston include cadmium, chromium, and silver. These metals may be present at levels that could cause EPA Land Disposal Restriction (LDR) treatment standards to be exceeded. Water and sludge in this pond also contain high concentrations (approximately 10 percent) of nitrates and other salts that may adversely affect solidification. Other constituents detected at high concentrations include potassium, sodium, and sulfate. There is a crust near the bottom of this pond that contains salt crystals. A silty sludge layer is present beneath the crust. An accurate solids content of the sludge is not available. Low concentrations (less than 1 ppm) of organics were detected during previous Weston analyses.

### **1.1.4 Clarifier**

The clarifier tank is located between Pond 207A and Pond 207C. The clarifier tank contains approximately 25,000 gallons of sludge and water derived from Pond 207A when the pond was being treated to generate pondcrete. Studies are currently underway to further refine the volume of, and to characterize, the waste contained in the clarifier. Limited previous analyses are available for clarifier wastes, but the chemical characteristics are expected to be similar to Pond 207A sludge.

### **1.1.5 Pondcrete**

There are approximately 8,000 blocks of pondcrete waste that do not



meet Nevada Test Site (NTS) waste acceptance criteria. These blocks were made by mixing Pond 207A sludge with portland cement. The pondcrete blocks are packaged in three-piece, heavy cardboard containers (triwalls) that have been banded for additional strength. The blocks weigh between 900 and 1,300 pounds. The majority are on wooden pallets. Free liquids have been observed for some of the existing triwall containers which resulted in the containers having been placed within larger sealed metal containers.

#### **1.1.6      Saltcrete**

There are approximately 2,500 saltcrete blocks that are packaged in triwalls that have been banded for reinforcement. The weight of each block is approximately 1,400 to 1,500 pounds. The majority are on wooden pallets. Some of the existing triwall containers have suffered weather decomposition or physical damage and have been placed within larger sealed metal containers. Additionally, saltcrete has been placed in half-crates which are approximately 2 x 4 x 7 foot wooden crates.

Large quantities of nitric acid used in the chemical processing operations at the plant produce a waste stream containing a high concentration of nitrate compounds. The waste stream is treated using precipitation, clarification, and evaporation. The effluent from the evaporator has ranged over the years from a 55 percent to a 35 percent (by weight) nitrate salt solution that is fed into a spray dryer and the dried salt is mixed with portland cement to produce the saltcrete waste form. Most of the constituents of the spray dryer are very soluble in water and are susceptible to water leaching and transport.

The triwalls contain a waste loading of 50 to 55 percent by weight and half-crates primarily contain a waste loading of 33 to 35 percent by weight. The metal containers store triwalls that have been damaged.

## **1.2 Project Purpose and Description**

The purpose of this treatability study is to test and evaluate various CSS formulations for the subject wastes. Candidate formulations will be selected that will be used to process the subject waste sources into final waste forms that meet all requirements currently in effect. These include requirements of U.S. Environmental Protection Agency (EPA) Region VIII, the Colorado Department of Health, U.S. Department of Transportation (DOT), and the Nevada Test Site (NTS), which is the facility that will dispose of the treated wastes. The final waste form must be certifiable and acceptable for storage, transportation, and land disposal.

The performance standards for the final solidified/stabilized waste form products, as defined in the overall project Scope of Work (Appendix 7), are as follows:

- Chemically and physically stable as defined by NVO-325, October 1988.
- A solid (no free liquids) as defined by NVO-325, October 1988, Section 2.2.2-D; DOT regulations in 49 CFR 173; EPA Test Method 9095 (EPA-246); and ASTM Method D4359-90.
- Transportable in interstate commerce as defined by DOT regulations in 49 CFR 173 and EPA regulations in 40 CFR 263.

- Within radioactive limits as defined by NVO-325, October 1988, Section 2.1.1-D and DOT Regulations in 49 CFR 173, Subpart I (173.400).
- Certifiable as a waste as defined by (1) the Rocky Flats Quality Assurance Manual, Low Level Waste Management Plan, 1-10000 EWQM, Section 1.1, RFP Procedures and (2) NVO-325, October 1988.

The specific final requirements and confirmatory test/inspection procedures for the final waste form product will be established during the development of the Process Control Plan (which is not within the scope of this treatability study).

NVO-325, Section 2.2.1-F contains the following language concerning waste stability requirements. "Where practical, waste shall be treated to reduce volume and provide a more physically and chemically stable waste form. If necessary, the waste shall be treated to assure that significant quantities of harmful gases, vapors, or liquids are not generated. Wastes shall not significantly react with packaging during normal storage, shipping, and handling times." By the nature of the proposed treatment (solidification), it will not be possible to reduce waste volume; however, the intent of treatment will be to produce a stable final waste form.

NTS waste form criteria for mixed waste disposal are contained in NVO-325, Section 2.2 and are summarized as follows:

- There must be no free liquids as determined by EPA Method 9095 [40 CFR 264.314(c)].

- Mixed waste that is prohibited from land disposal will not be accepted unless treated as specified in 40 CFR 268, Subpart D (Treatment Standards).

NTS waste package criteria are contained in NVO-325, Section 2.1 and are summarized as follows:

- The waste package must be in accordance with all applicable DOT regulations.
- The waste package must have an external radiation level of  $\leq 200$  mrem/hr on contact.
- The waste package strength must be able to support 4,000 lb/ft<sup>2</sup> (28 psi).
- The weight limit for each package is 9,000 pounds, unless special arrangements are made.

During the treatability study, numerous tests and analyses will be performed on treated waste forms at various phases of the study to verify compliance with final waste form criteria.

Unconfined compressive strength (UCS) tests will be conducted, and the results will be used as a screening tool for comparing the success of various formulations.

The RCRA Land Disposal Restriction (LDR) treatment standards for the EPA Hazardous Waste Numbers associated with these wastes included treatment standards for metals, cyanide, and organics (see Table 1-1). Based on previous waste characterization efforts, the concentrations of cyanide and organics detected are not expected to exceed the LDR treatment standards. This will be confirmed during

**TABLE - 1-1**  
**LDR TREATMENT STANDARDS - PONDCRETE/SALTCRETE**  
**ROCKY FLATS**

REGULATED HAZARDOUS CONSTITUENT	LDR TREATMENT STANDARD (NONWASTEWATERS) <sup>(1)</sup>		
	F001-F003, F005	F006, F007, F009	D006 <sup>(2)</sup>
Acetone	160 mg/kg <sup>(3)</sup>	NA	NA
n-Butyl alcohol	2.6 mg/kg <sup>(3)</sup>	NA	NA
Carbon tetrachloride	5.6 mg/kg <sup>(3)</sup>	NA	NA
Chlorobenzene	5.7 mg/kg <sup>(3)</sup>	NA	NA
Cyclohexanone	0.75 mg/L <sup>(4)</sup>	NA	NA
1,2-Dichlorobenzene	6.2 mg/kg <sup>(3)</sup>	NA	NA
Ethyl acetate	33 mg/kg <sup>(3)</sup>	NA	NA
Ethylbenzene	6.0 mg/kg <sup>(3)</sup>	NA	NA
Ethyl ether	160 mg/kg <sup>(3)</sup>	NA	NA
Isobutanol	170 mg/kg <sup>(3)</sup>	NA	NA
Methanol	0.75 mg/L <sup>(4)</sup>	NA	NA
Methylene chloride	33 mg/kg <sup>(3)</sup>	NA	NA
2-Butanone (MEK)	36 mg/kg <sup>(3)</sup>	NA	NA
4-Methyl-2-pentanone (MIBK)	33 mg/kg <sup>(3)</sup>	NA	NA
Pyridine	16 mg/kg <sup>(3)</sup>	NA	NA
Tetrachloroethene (PCE)	5.6 mg/kg <sup>(3)</sup>	NA	NA
Toluene	28 mg/kg <sup>(3)</sup>	NA	NA
1,1,1-Trichloroethane	5.6 mg/kg <sup>(3)</sup>	NA	NA
1,1,2-Trichloro-1,2,2-trifluoroethane	28 mg/kg <sup>(3)</sup>	NA	NA
Trichloroethene (TCE)	5.6 mg/kg <sup>(3)</sup>	NA	NA
Trichlorofluoromethane	33 mg/kg <sup>(3)</sup>	NA	NA
Xylene	28 mg/kg <sup>(3)</sup>	NA	NA
1,1,2-Trichloroethane	7.6 mg/kg <sup>(3)</sup>	NA	NA
Benzene	3.7 mg/kg <sup>(3)</sup>	NA	NA
2-Nitropropane	Incineration <sup>(5)</sup>	NA	NA
2-Ethoxyethanol	Incineration <sup>(5)</sup>	NA	NA
Cyanides (Total)	NA	590 mg/kg <sup>(3)</sup>	NA
Cyanides (Amenable)	NA	30 mg/kg <sup>(3)</sup>	NA
Cadmium	NA	0.066 mg/L <sup>(4)</sup>	1.0 mg/L <sup>(4)</sup>
Chromium (Total)	NA	5.2 mg/L <sup>(4)</sup>	NA
Lead	NA	0.51 mg/L <sup>(4)</sup>	NA

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TABLE - 1-1  
LDR TREATMENT STANDARDS - PONDCRETE/SALTCRETE  
ROCKY FLATS  
PAGE 2 OF 11

REGULATED HAZARDOUS CONSTITUENT	LDR TREATMENT STANDARD (NONWASTEWATERS) <sup>(1)</sup>		
	F001-F003, F005	F006, F007, F009	D006 <sup>(2)</sup>
Nickel	NA	0.32 mg/L <sup>(4)</sup>	NA
Silver	NA	0.072 mg/L <sup>(4)</sup>	NA

(1) Wastewaters are defined by 40 CFR 268.2(f) as wastes that contain less than 1% TOC and 1% TSS by weight. Also, for F001-F005 solvent mixtures, wastewaters must contain less than 1% total F001-F005 solvents, by weight.

(2) Applies only to pondcrete.

(3) Concentration in waste (CCW).

(4) Concentration in waste extract (CCWE).

(5) Specified treatment technology.

LDR - Land Disposal Restrictions, 40 CFR Part 268.

NA - Not applicable.

the waste characterization currently underway. During certain phases of the treatability studies for the various waste forms, treated wastes will be leached using the Toxicity Characteristic Leaching Procedure (TCLP) method and analyzed for metals. During final testing, compliance with all applicable LDR treatment standards will be verified, including cyanide and organics if required. However, for costing purposes, it has been assumed that neither cyanide nor the organics of concern will be detected at sufficient concentrations in the raw waste forms, such that further testing for these compounds in the stabilized waste forms will not be required.

During the final phases of testing, treated wastes will be tested by the Paint Filter Liquid Test (EPA Method 9095 in SW-846) to verify that there are no free liquids; however, the presence of free liquids is not expected to be a problem because of the nature of the treatment process. Also, the treated wastes will be tested by the Liquid/Solid Test (ASTM D4359-90, determining whether a material is a liquid or a solid) for compliance with DOT regulations.

Treated wastes will be tested for durability and stability using wet/dry and freeze/thaw resistance tests. For Pond 207C waste and saltcrete, this is especially important because, historically, the saltcrete has been subject to weather decomposition. It is suspected that salt crystals grew in the saltcrete waste form, causing it to effloresce, expand and fail.

These durability tests are currently included within the treatability study work plan since the NTS is currently not capable of receiving waste and no alternate location has been selected.

### 1.3 Scope of Work

Treatability studies will be performed at the HALLIBURTON NUS Laboratory in Pittsburgh, Pennsylvania. This laboratory is

licensed (NRC License No. 37-17937-03) to handle radioactive material and has the facilities and personnel required to perform the scope of work outlined herein.

### 1.3.1 Goals and Objectives

The goals of these treatability studies (for pond sludge, pondcrete, saltcrete, and clarifier sludge) are to develop process design parameters for pre-processing waste sources and to develop chemical solidification and stabilization formulations for each waste source. CSS formulations will be developed for the following waste forms:

- Pond 207A sludge
- Pond 207B sludges
- Clarifier sludge and water
- Pond 207C slurry (sludge and water mixed)
- Pondcrete
  - Triwalls
  - Triwalls in metal containers
- Saltcrete
  - Triwalls
  - Half-crates
  - Triwalls in metal containers

Prior to the beginning of full-scale treatment, EG&G has stated that they will evaporate as much water as possible from Pond 207A and from one of the three 207B-series ponds. The pond water will be stored in the other two ponds. The sludge will be sized to 10 mesh during the consolidation process. As part of this preprocessing, the existing clarifier and other sizing and screening equipment will be used to separate pond sludge and water. Sludge that is present in the existing clarifier will be removed by



EG&G and placed into drums so that the clarifier can be repaired.

As part of the treatability study, the gravity settling characteristics of 207A and 207B pond sludges will be evaluated. This information is needed to determine whether the existing clarifier is adequate and whether an additional or different clarifier is needed for pre-processing pond sludge during consolidation activities. Bulk settling rate tests will be conducted to determine the bulk rise rate of sludge and the required clarification overflow rate. Jar tests will also be conducted to evaluate the need for coagulant addition and the optimum coagulant dosage(s).

In the full scale treatment, pondcrete and saltcrete will be slurried for treatment and trash extrusion, sized and ground to 10 mesh, and mechanically dewatered prior to the addition of CSS binders and admixtures. The trash (wooden pallets, cardboard and plastic) will be ground to 10 mesh and will be incorporated at 8% of the mixture (4% - 5% of the final product). Pond sludge will be sized and ground to 10 mesh and mechanically dewatered prior to the addition of CSS binders and admixtures. Dewatering tests will be conducted on each waste form to determine the necessary design parameters and the achievable cake solids.

The CSS formulation portion of the treatability study will constitute the majority of the laboratory work. The objectives of these studies and experiments include the following:

- Determine critical physical variables that affect CSS formulations and performance.
- Determine the optimum ratios for waste form to binder(s) to admixture(s) to achieve acceptable physical

characteristics and chemical leachability criteria for each waste form.

- Develop the statistical basis for optimum formulation application.
- Evaluate long-term storage characteristics (durability) of the final treated waste forms.

A multilevel factorial experiment design will be used to evaluate the interaction of parameters such as the following:

- Binders (e.g., portland cement, fly ash, lime).
- Additives/admixtures (e.g., latex, silica, plastic fibers).
- Water content.

Because of the number of variables that affect the development of successful CSS formulations (recipes), factorial experimental designs will be required during recipe development. Measured responses will be evaluated as a function of the variable to determine optimum responses and key variable interactions.

Factorial experiments are characterized in that the effect of changes on one variable can be assessed independently of the other variables. The factorial experiment is accomplished by using, as the design, each of the possible combinations of the levels (concentrations) of each factor (parameter or variable). In a factorial experiment, all factors may be varied simultaneously. The factorial approach allows the assessment of the interaction of two or more variables.

The general concept used for developing process formulations for the various waste forms follows a progression from screening binder/waste formulations through a more comprehensive evaluation of variables and additives to regulatory compliance testing of candidate formulations that passed all of the previous evaluation criteria.

The screening tests will use an accelerated curing procedure where various ratios of waste, binder, additives, and water are prepared and cured at elevated temperatures for 48 hours. Formulations that do not develop sufficient strength, contain excess water, or fail assigned acceptance criteria will be eliminated from further consideration.

The accelerated curing procedure provides an indication of the potential strength and durability of solidified waste. It is used as an indicator of the probability that the desired compressive strength and durability can be obtained by use of a particular CSS formulation. This procedure is used to save time relative to allowing the test specimens to cure for the conventional 28-day period.

Accelerated curing tests will be used for all phases of the treatability studies except for the regulatory compliance testing. A modified version of ASTM Method C684-89 (Procedure A - Warm Water Method) will be used for accelerated curing of test specimens (see Section 7.0 for additional details). The ASTM method will be modified to allow for a 48-hour cure rather than the 24-hour cure specified in the method. The size of the test specimen molds to be used will also vary from the ASTM method. After curing, products will be tested for UCS using ASTM Method ASTM D4289-83. TCLP extraction and metals analysis will also be conducted on some specimens. The freeze/thaw (ASTM D560-89) and wet/dry (ASTM

D559-89) durability test procedures will be modified to enable the testing to be completed prior to the start of the final phase. The control cylinder (i.e., volume and moisture loss specimen) will be omitted; thus, only one cylinder (i.e., weight loss specimen) will be submitted for each test. The dimension measurements and the weighing of the cylinders are to be omitted. Brushing of the cylinders shall be done as specified in the methods. The freeze/thaw procedure will be accelerated by reducing the time of freezing from 24 hours/cycle to 12 hours/cycle. The wet/dry procedure will be accelerated by decreasing the drying period from 42 hours to 19 hours and the time of submergence from 5 hours to 4 hours. Using these accelerated methods, testing will be completed in time to incorporate the results in the final phase.

The test results from the specimens cured by the accelerated method will allow for rapid assessment of the variability of various CSS formulations. Tests performed on specimens subjected to accelerated curing will be used as a screening tool to determine whether specific CSS formulations should be subjected to additional testing or screened from further evaluation.

Final testing to determine compliance with regulatory and disposal site requirements and to determine long-term durability will be performed on specimens that have been allowed to cure using the conventional 28-day procedure.

Laboratory procedures for mixing, curing, testing, and physical and chemical analysis of test specimens are provided or referenced in Section 6.0.

The following sections provide details on the treatability study experiments to be used for the various waste forms that are to be included in the laboratory testing.

### **1.3.2 Pond 207A Sludge, Pond 207B Sludge, and Clarifier Sludge**

#### **1.3.2.1 Assumptions**

The scope of the treatability studies for these waste forms will be the same because the wastes are similar in nature based on previous analysis.

Prior to treatability testing, the sludges will be screened to 10 mesh size. Material that does not pass a 10 mesh sieve will be ground to 10 mesh size, if this can be done in a timely manner that will not adversely affect the schedule. Otherwise, treatability testing will be performed on material that passes 10 mesh. During previous grain size analysis by Weston, the majority of the sludge particles was determined to be less than 10 mesh size. Formulations using material that passes 10 mesh and material "as received" will both be tested to determine whether particle size has any affect on the CSS formulation. Based on existing data that shows a relatively small percentage of the solids greater than 10 mesh, and observation of the sludge indicating that most of the large solids are inert, it is assumed that particle size will have little or no effect on CSS formulation.

Portland (Type I and Type V) cement will be evaluated as the main additive for these sludges. Portland cement-based treatment has worked in the past during original processing of the solar pond sludge and during reprocessing of pondcrete blocks that failed NTS disposal site acceptance criteria. Lime addition will be evaluated only if it is needed in order to maintain an alkaline pH needed to minimize the solubility of heavy metals.

The preliminary treatment process design assumes that the sludges will be mechanically dewatered prior to mixing with portland cement

and water. Therefore, to simulate anticipated process conditions, sludge samples will be mechanically dewatered before beginning treatability study work. Limited dewatering tests may be performed during the preliminary test phase to determine the percent solids achievable.

The addition of additives to enhance durability (e.g., fibers, air entrainment, and silica) will take place only after successful CSS formulations have been identified.

The treatability study schedule assumes the following:

- Assume B Ponds will be consolidated by EG&G.
- Accelerated curing of test specimens will be required in order to meet schedule constraints. Final testing to determine compliance with regulatory requirements and to verify long-term durability will be conducted on specimens that have cured for 28 days.
- Twelve batches (recipes) can be mixed per day (one shift).
- Two to four unconfined compressive strength (UCS) tests can be conducted per hour.
- The Toxicity Characteristic Leaching Procedure (TCLP) metals analytical results will be available in 7 days, based on a maximum of 10 samples extracted per day and low activity of the samples.
- The results for full TCLP analysis (organics, if required, and inorganics) will be available in 6 weeks

(maximum) .

- Wet/dry and freeze/thaw durability testing will take 24 days to complete.

#### 1.3.2.2 Preliminary Testing

As part of the characterization study performed on the clarifier sludge, amenable cyanide will be done per EPA 335.1. This will determine if the clarifier sludge will meet the LDR requirements stated in 40 CFR 268 for F006 classified waste.

The dry solids content of each sludge will be determined.

The sludges will be screened to 10 mesh size. Oversize material (greater than 10 mesh) will be ground to 10 mesh, if this is possible without affecting the schedule.

Quick screening mixing tests will be performed on each of the sludges. This will involve mixing portland cement and water, if needed, with the sludges to obtain a "workable" mixture that, in the opinion of the laboratory technician, is neither too wet nor too dry. The initial starting point will be based on the experience of the laboratory technician, past experiences at Rocky Flats, and mixtures provided in literature for similarly characterized wastes. The ratios of waste, portland cement, and water that are determined to be the best mixes based on this preliminary testing will be used as the starting points (center points in factorial experiments) for the next phase of the treatability study.

#### 1.3.2.3 Phase I

This phase involves an experiment using sludge, portland cement, and water for each of the three sludges. The experiment will bracket the center points determined in the preliminary testing. That is, the sludge concentration will be held constant, and the portland cement and water content will each be varied by a fixed percentage above and below the center point. The percent variation in the amount of portland cement and water added will also be based on the preliminary testing. This means that, for each sludge, three portland cement concentrations and three water concentrations will be evaluated. For example, the center point could be 3 parts waste, 1 part Portland cement, and 1 part water. Then, formulation would be developed using 3 parts waste,  $1 \pm$  parts Portland cement, and  $1 \pm y$  parts water. This will result in five recipes for each of the three sludges (see Figure 1-2).

After the specimens for each formulation are mixed and placed into plastic test cylinders, they will be cured for 48 hours using the accelerated method. After curing, visual observations on free water content and the appearance of the specimens will be made.

Formulations that pass the visual inspection will be subjected to UCS testing. Formulations that do not achieve sufficient strength will be eliminated from further evaluation.

#### 1.3.2.4 Phase II

Phase II will basically repeat the Phase I testing. This phase of the treatability study will also evaluate testing of 10 mesh sized sludges versus "as received" sludges. The recipes will be adjusted, as needed, to evaluate the acceptable range of mixes and



VARIABLE #2  
(e.g. WATER)

VARIABLE #1  
(e.g. PORTLAND CEMENT)

LEGEND

- \* CENTER POINT CONCENTRATION
- CONCENTRATIONS  $\pm$  %  
AROUND CENTER POINT

FIGURE 1-2

EXAMPLE OF TWO VARIABLES EVALUATED  
AT THREE CONCENTRATIONS  
ROCKY FLATS, GOLDEN, COLORADO  
NOT TO SCALE



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to define the new center point, which would be the best anticipated recipe based on strength. Each formulation will use both 10 mesh sized sludges and "as received" sludges. This will result in ten recipes for each of the three sludges. After the specimens for each formulation are mixed and allowed to cure for 48 hours (accelerated method), they will be evaluated for strength.

Formulations that pass the strength criteria will be leached using the TCLP method, and the extracts will be analyzed for metals. Formulations that do not achieve the Land Disposal Restriction (LDR) treatment standard for metals of concern may be eliminated from further consideration.

The pH of the TCLP extract will also be measured to determine whether the addition of lime (calcium hydroxide) is needed to maintain minimum solubility of the metals. The premise for this experiment is that if the pH of the TCLP extract is too low, there is insufficient buffering capacity of the solidified waste, and lime addition would be needed to maintain a high enough pH to maintain minimum metals solubility. The amount of lime required will be based on a titration-type of test and/or stoichiometry.

#### 1.3.2.5 Phase III

Recipes, using portland cement (or portland cement and lime, if required) and using 10 mesh sized sludge or "as received" sludge, that exhibit the best strength and leachability characteristics will be tested in this phase. This phase of the treatability study will also evaluate the addition of admixtures (e.g., plastic fibers, air entrainment, and silica), long-term durability (i.e., wet/dry and freeze/thaw resistance), and regulatory compliance testing.

For each sludge, four potential recipes will be evaluated. These will include four recipes to evaluate admixtures (three admixtures and one recipe without admixtures). Duplicate specimens will be prepared for the proposed tests, except for wet/dry and freeze/thaw resistance, where the tests use a control specimen.

During this phase, TCLP analysis will be conducted for the selected CSS Formulation on a wet mix (i.e., less than 1 hour after the mixture is prepared). These results will be compared with TCLP Analysis after a 28-day cure. This will be done to verify that TCLP metals concentrations will not fluctuate in an adverse manner from initial preparation to after a 28-day cure time. These results will be beneficial during actual remediation, thereby suggesting that samples collected during the initial pouring will indicate whether the billet will meet LDR requirements.

Additionally, two cylinders prepared with the selected CSS Formulation will be cured for two days in a moist atmosphere, simulating expected conditions during remediation. These cylinders will be tested for UCS. The results will be compared to the 28-day UCS test results thereby providing a strength requirement after two days of curing. This data will provide a way to determine acceptability of a billet during remediation.

After mixing, the specimens will be allowed to cure for 7 days and evaluated for strength (UCS) and metals leachability (TCLP). For mixes where strength and leachability are acceptable, specimens will be allowed to cure for 28 days total. After the 28-day cure, the specimens will be evaluated for strength, compliance with LDR treatment standards (TCLP and analysis for inorganics and organics of concern, if required), and durability (wet/dry and freeze/thaw resistance). Specimens that have been subjected to wet/dry and freeze/thaw tests will be also evaluated for strength following

these durability tests to determine whether a decrease in strength has occurred.

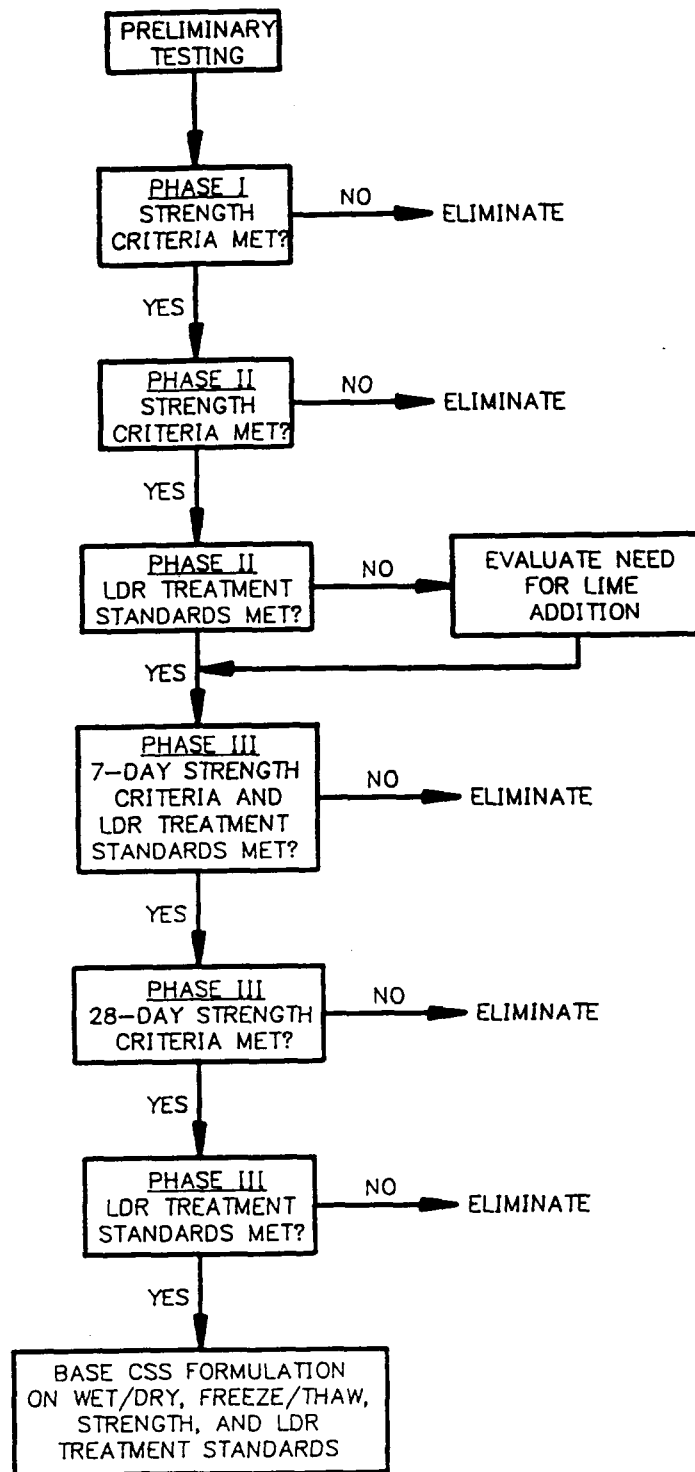
A petrographic analysis will also be performed for each formulation to determine the microscopic structure of the solidified wastes.

Based on the Phase III results, a CSS formulation for each sludge will be selected for use in full scale treatment.

A decision tree showing the CSS formulation screening and selection process for these sludges is presented in Figure 1-3. A summary of the treatability study for these sludges is presented in Table 1-2.

#### **1.3.3      Pondcrete**

The treatability study for pondcrete will consist of three phases. A conceptual outline for the three phases is provided in Appendix H. Phase I will include physical tests and other testing necessary to determine major process options, thus allowing early selection of equipment. Phase I will consist of two sub-phases (IA and IB). Phase IA will consist of tests to determine the upper limit of waste loading and other engineering parameters such as viscosity, bulk density, specific gravity, etc. Phase IB will consist of dewatering studies and tests to evaluate the affect of inclusion of various amounts of trash. Phase II will consist of testing to screen various cement stabilization and solidification (CSS) formulas for key parameters, including any additives needed for compliance with land disposal restrictions (LDRs). Phase II will also include accelerated durability and UCS testing. Phase III will consist of testing to determine appropriate operating ranges for key process parameters and to demonstrate full regulatory compliance. Phase III will also include additional studies for miscellaneous concerns such as the affect of different curing temperatures and addition of a "superplasticizer". The results



**CSS FORMULATION SCREENING  
AND SELECTION PROCESS  
POND 207A, POND 207B,  
AND CLARIFIER  
ROCKY FLATS, GOLDEN, CO**

**FIGURE 1-3**



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TABLE 1-2  
POND 207A SLUDGE, POND 207B SLUDGE, CLARIFIER SLUDGE  
ROCKY FLATS

Phase	CSS Binder	Additives	# of Mixes per Sludge	Cure Time	Test	# of Test per Sludge	Duration	Comments
Preliminary	None	None	NA	NA	Dry solids	1		1 week total duration.
	None	None	NA	NA	Size of 10 mesh	1		
	Portland Cement	None	TBD	NA	Visual	TBD		
Phase I	Portland Cement	None	5	2 days	UCS	5	1 week	Duration: 1 week total. Goal: Bracket center point of binder formulations based on strength and free liquid content.
Phase II	Portland Cement	None	5	2 days	UCS	5	1 week	Duration: 2 weeks total. Perform tests on 10 mesh and as received sludges.
					TCLP metals	5	1 week	Goal: Define center point of mixtures based on strength and leachability of metals.
					TCLP metals	5	1 week	
Phase III	Portland Cement	None	1	None (1)	TCLP metals	1	1 week	Duration: 10+ weeks total. Goal: Define mixture of ratio of binder + additives to meet all LDR requirements, and long term durability.
				2 Days (2)	UCS	2	1 week	
				7 Days	UCS, TCLP metals	1	2 weeks	
				28 Days	UCS, PFLT, TCLP, wet/dry and UCS, freeze/thaw & UCS	1	10 weeks	

TABLE 1-2  
POND 207A SLUDGE, POND 207B SLUDGE, CLARIFIER SLUDGE  
ROCKY FLATS  
PAGE 2 OF 3

Phase	CSS Binder	Additives	# of Mixes Per Sludge	Cure Time	Test	# of Test Per Sludge	Duration	Comments
Phase III (Cont'd)	Portland Cement	Plastic Fibers	1	None (1)	TCLP Metals	1	1 week	
				2 days (2)	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	
				28 days	UCS PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	
	Portland Cement	Air Entrainment	1	None (1)	TCLP Metals	1	1 week	
				2 days (2)	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	
				28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	
	Portland Cement	Silica Flour	1	None (1)	TCLP Metals	1	1 week	
				2 days (2)	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	
				28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	
				2 days (2)	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	
				28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	

TABLE1-2  
POND 207A SLUDGE, POND 207B SLUDGE, CLARIFIER SLUDGE  
ROCKY FLATS  
PAGE 3 OF 3

CSS - Chemical Stabilization and Solidification  
TBD - To Be Determined Based On Visual Observations  
UCS - Unconfined Compressive Strength  
TCLP- Toxicity Characteristic Leaching Procedure  
PFLT- Paint Filter Liquid Test

- (1) To be performed less than 1 hour after mixing.
- (2) Air dry curing.



from preliminary phases may cause subsequent tests to be revised from that contained in this work plan.

The pondcrete testing will be performed using both triwalls and triwalls in metal containers unless otherwise noted.

#### 1.3.3.1 Assumptions

Two types of pondcrete (from triwalls and from triwalls in metal containers) will be subject to testing because the materials vary in consistency.

The solids content of the pondcrete will be analyzed to determine and calculate dry solids. The amount of pozzolanic binders needed will be based on the dry solids of the pondcrete. The fluidity limit will be determined for pondcrete. This will dictate how much water must be added for the process stream.

The preliminary treatment process design assumes that the pondcrete will be crushed so that all material is below a 10 mesh size, slurried to remove the oversize material, and then mechanically dewatered prior to mixing with the selected binder and water. Dewatering tests will be performed by vendors to determine the achievable sludge cake solids. CSS formulations for pondcrete will be based on dry solids; therefore, necessary adjustments to binders, additives, and water to be added under full scale conditions can be made based on these results.

Pondcrete samples will be ground/milled to 10 mesh screen size during field sampling, so size reduction will not be needed during the treatability study work conducted in the Pittsburgh Laboratory.

The addition of additives to enhance durability will take place

only after successful CSS formulations have been identified.

The treatability study schedule assumes the following:

- Accelerated curing of test specimens will be required in order to meet schedule constraints. Final testing to determine compliance with regulatory requirements and to verify long-term durability will be conducted on specimens that have cured for 28 days.
- Twelve batches (recipes) can be mixed per day (one shift)
- Two to four UCS tests can be conducted per hour.
- The TCLP metals analytical results will be available in 7 days, based on a maximum of 10 samples extracted per day and low radioactivity of samples.
- The results for full TCLP analysis (organics, if required, and inorganics) will be available in 6 weeks (maximum).
- Wet/dry and freeze/thaw durability testing will take 24 days to complete.

#### 1.3.3.2 Phase IA

Phase IA consists of initial analytical testing and engineering studies.

#### Initial Analytical Testing of Feed Waste

Initial testing of the pondcrete waste forms includes a methanol

study, baseline chemical analysis, and analysis for engineering parameters.

### 1. Methanol Study

The goal of the methanol study is to determine whether methanol is present in triwalls in metal containers and, if so, whether it could be expected to leach at concentrations that exceed LDR treatment standards. Based on previous characterization data, the presence of methanol above LDR treatment standards is not a concern for triwalls. A total methanol analysis will be conducted using a GC method on a sample from the triwalls in metal containers. If methanol is detected, its presence will be confirmed using a second GC column. A zero head space (ZHE) TCLP extraction will also be conducted on samples spiked at two methanol concentrations (average and twice the average concentration). The extract will be analyzed to determine whether methanol will leach at a concentration that exceeds the LDR treatment standard.

### 2. Baseline Analysis

The baseline analysis will determine baseline chemical data for comparison with characterization data. Representative samples of both triwalls and triwalls in metal containers will be analyzed for a wide range of parameters, including total metals, leachable metals, radionuclides, anions, cations, and other analytes (see Table 1-3).

### 3. Engineering Parameters

Representative samples of triwalls and triwalls in metal containers will be analyzed for bulk density, moisture content (gravimetric and Karl Fisher percent water), specific gravity, and specific

**TABLE 1-3**  
**INITIAL ANALYTICAL TESTING SUMMARY**  
**PONDCRETE/SALTCRETE TREATABILITY STUDY**  
**ROCKY FLATS**

Analytes	
<u>Metals</u> Aluminum Arsenic Antimony Barium Beryllium Boron Cadmium Calcium Chromium, total Cobalt Copper Iron Lead Magnesium Manganese Mercury Nickel Potassium Selenium Silicon Silver Sodium Strontium Thallium Tin Vanadium Zinc  <u>Radionuclides</u> Americium (Am-241) Plutonium (Pu-239/Pu-240)	<u>Others</u> Alkalinity, carbonate <sup>(1)</sup> Alkalinity, Methyl Orange <sup>(1)</sup> Alkalinity, phenolphthalein <sup>(1)</sup> Ammonia Chloride <sup>(1)</sup> Cyanide (total and amenable) Fluoride <sup>(1)</sup> Nitrate <sup>(1)</sup> pH Phosphorus, total <sup>(1)</sup> Specific conductance <sup>(1)</sup> Sulfate <sup>(1)</sup> Sulfide <sup>(1)</sup> Total organic carbon  <u>Leaching Tests</u> TCLP Leach - Arsenic - Barium - Cadmium - Chromium, total - Lead - Nickel - Selenium - Silver  ASTM Leach - Chloride - Nitrate - Phosphorus, total - Sulfate - Total dissolved solids

(1) Following dissolution in deionized water.

gravity of discrete particles. These analyses will determine the baseline for engineering parameters.

### Engineering Studies

Engineering studies to be conducted during Phase IA include a waste loading study, a dissolution test in water, a determination of viscosity and density of samples at various solids contents, settling tests, an evaluation of the rheology of slurries, and an evaluation of saturation total dissolved solids (TDS) versus temperature.

#### 1. Waste Loading Study

The goal of the waste loading study is to determine the upper limit of the waste loading (% feed waste in the product slurry), which will help to determine the degree of dewatering that will be necessary (i.e., if 20 percent was the upper waste loading, then it would not be necessary to dewater to more than 20 percent solids). The results of this testing would narrow the selection of downstream process options.

The waste loading study will consist of mixing the two waste forms, at different solids contents, with the CSS formulation used for the solar ponds. The CSS formulation will consist of Type V portland cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.75, respectively. The waste loading will vary based on the total solids content of the waste. Mixes will be prepared at solids loadings of 20, 30, 40, 50, 60, and 70 percent. The liquid phase, with the solids concentration at the saturation point (as determined by the dissolution test), will also be solidified using the same CSS formulation. All mixes will be prepared at a water-to-pozzolan ratio of 0.42. To assure uniform moisture content,

enough material for all batches will be prepared at the various solids loadings and stored in a sealed container until it is time to mix the waste material with the pozzolans.

Analysis of the input (i.e., prior to mixing with pozzolans) will include total solids, total dissolved solids of the supernatant, viscosity, specific gravity, and bulk density. Analysis of the output (i.e., after mixing but prior to curing) will include bulk density, VG Fann testing, and observation of the ability of the material to be pumped (this testing will be filmed).

After the specimens for each solids loading are mixed and placed into plastic cylinders, they will be cured for 48 hours using the accelerated curing method. After curing, visual observation of the free water content and appearance of the specimens will be made. Formulations that pass the visual inspection will be subjected to UCS, TCLP metal analysis, and accelerated durability testing (i.e., wet/dry and freeze/thaw resistance). Specimens that have been subjected to durability testing will also be evaluated for strength to determine whether a decrease in strength has occurred. Formulations that do not achieve sufficient strength or fail LDR requirements will be eliminated from further evaluation.

## 2. Dissolution Tests

The goal of these tests is to determine the extent of feed waste dissolution when mixed with water. This test consists of dissolving a known mass of sample in excess water and determining the specific gravity and TDS of the solution. The dry weight of the undissolved portion will also be determined.

Another test will consist of dissolving the sample in a small amount of water to make a saturated solution. The TDS and specific

gravity of the supernatant will be measured.

Both of these tests will be conducted at room temperature and 100°F.

### 3. Viscosity and Density at Various Solids Contents

The goal of these tests is to determine how the solids content affects the viscosity and density of the feed wastes. Various mixtures of the waste and water will be prepared at concentrations of 5, 10, 20, 30, 40, 50, and 60 percent solids (or up to the plastic limit). The viscosity and specific gravity will be measured for each mixture. The viscosity of the supernatant, up to the saturation point, will also be determined. The experiment will be performed at room temperature and repeated at 100°F to reflect the temperature rise expected in the grinding circuit.

### 4. Settling Tests

Settling tests will be conducted on pondcrete samples ground to 10 mesh to determine settling rates and terminal densities. These tests will be performed using a saturated solution as the liquid phase and will be conducted at solids concentration of 5, 10, 20, 30, 40, 50, and 60 percent. The tests will be performed at room temperature and at 100°F.

### 5. Evaluation of Rheology of Slurries

For these tests, the viscosity of slurries will be measured at solids contents of 5, 10, 20, 30, 40, and 50 percent, of feed waste, with and without trash included in the slurry. This testing will be conducted on a saturated solution. Each slurry with trash will include 20 percent dry weight of trash (i.e., pallet, plastic

sheeting, steel, etc.). The "steel" material will include steel banding, nails and artifacts from the ball mill. The trash comprises approximately 8 percent of the total weight of a billet. When calculated using dry weights, approximately 20 percent of the billet is considered to be trash. The viscosity of the slurry is needed to define pumping, settling, and other material handling parameters.

## 6. Saturation TDS versus Temperature

The goal of these tests is to measure the degree of salt dissolution at various temperatures. This will be determined by collecting samples of the supernatant at different temperatures (i.e., room temperature, 50°F, and 100°F). The samples will be analyzed for TDS and specific gravity.

### 1.3.3.3 Phase IB

This portion of Phase I will consist of evaluating dewatering processes and performing a trash study. The dewatering study will evaluate the selected process option that is believed to be the most practical to achieve the optimum solids content determined in the waste loading study (Phase IA). The trash study will evaluate various loading of trash to determine whether there is any impact from different loadings.

## 1. Dewatering Study

This testing will evaluate various dewatering devices that are capable of achieving the solids content derived in the waste loading study. Vendors will be solicited to conduct the dewatering tests. Dewatering testing will include the appropriate quantities of trash. Further details on these tests will be developed at the



completion of the waste loading study.

## 2. Trash Study

This study will evaluate trash addition with regard to chemical and physical parameters. Testing will be conducted at the maximum solids content determined from the waste loading study. Trash will be added at concentrations of 2, 5, 7.5, 10, 12.5, 15, 20, 50, 75, and 100 percent of the feed waste loading. The objective of these tests is to eliminate trash as a future variable for the CSS formulation development to be conducted during Phase II.

After the samples are mixed with various percentages of trash, they will be tested for percent solids, bulk density, viscosity, specific gravity, and percent water (Karl Fisher).

CSS testing will be conducted to determine the effect of trash on the stability of the solidified product and TCLP criteria. The CSS testing will be conducted at three waste loadings. One of the waste loadings will be that selected from the initial waste loading study. The others will bracket either side of the initial loading (e.g., plus or minus 10 percent solids). The water-to-pozzolan ratio will be 0.42. The CSS formulation will consist of Type V portland cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.75, respectively. After the specimens for each formulation are mixed and placed into plastic test cylinders, they will be cured for 48 hours using the accelerated method. After curing, visual observations of free water content and the appearance of the specimens will be made. Formulations that pass the visual inspection will be subjected to UCS, TCLP for metals, and accelerated durability testing (i.e., wet/dry and freeze/thaw resistance). Specimens that have been subjected to durability testing will also be evaluated for strength to determine whether a

decrease in strength has occurred. Formulations that do not achieve sufficient strength or fail LDR requirements will be eliminated from further evaluation.

#### 1.3.3.4 Phase II - CSS Formulation Development

This phase will be conducted using only triwalls. Triwalls in metal containers will not be used unless concerns with methanol still exist based on the methanol study from Phase IA. Triwalls have the same contaminants as those that exceeded LDR treatment standards for the triwalls in metal containers but at higher concentrations.

These studies will be conducted at the solids content selected from the waste loading study. The water-to-pozzolan ratios will be varied and will include ratios of 0.34, 0.42, and 0.50. The CSS formulation will consist of Type V portland cement, Type C flyash and hydrated lime at a ratio of 1.0/2.0/0.75, respectively. This testing will determine whether additives are needed to improve the characteristics of the solidified product so that it will pass the TCLP testing criteria.

The product slurry produced from these tests will be subjected to viscosity (VG Fann) and density (mud balance) testing. The VG Fann testing will yield gel strength characteristics from the product slurries produced. The mud balance testing will determine the densities of the product slurries. Together these tests will provide the necessary data to engineer the product slurry handling systems.

After the specimens for each formulation are mixed and placed in plastic test cylinders, they will be cured for 48 hours using the accelerated method and analyzed for TCLP metals. If the TCLP results pass the LDR treatment standards, then Phase II will be

complete. If not, further testing will be required to evaluate various additives (to be determined) to reduce the leachability of the metal constituents.

Testing in this phase will be dependent on the results of the waste loading study. Upon completion of the waste loading study, additional information will be available to thoroughly scope the CSS formulation development phase.

#### **1.3.3.5 Phase III - Regulatory Compliance Testing**

Phase III will test the CSS formulation to determine whether it will pass all regulatory criteria over the proposed operating range. The proposed operating range will be at a water-to-pozzolan ratio of 0.34 to 0.50, with 0.42 being the center point. These water-to-pozzolan ratios will be tested at  $\pm 10$  percentage points around the waste loading selected from the Phase IA testing.

After mixing the product slurries will be subjected to VG Fann and mud balance. The specimens will be allowed to cure for 7 days and evaluated for strength (UCS) and metals leachability (TCLP). For formulations where strength and leachability are acceptable, the specimens will be allowed to cure for 28 days total. After the 28-day cure, the specimens will be evaluated for strength, metals leachability (TCLP) for compliance with LDR treatment standards, free liquid (Paint Filter Liquid Test and Solid/Liquid Test), and durability (i.e., wet/dry and freeze/thaw resistance). Specimens that have been UCS and TCLP metals testing to durability testing will also be evaluated for strength to determine whether a decrease in strength or an increase in the leaching metals has occurred.

Several additional tests will be performed during Phase III. A factorial experiment that varies the ratio of pozzolans will be

conducted so that the cement to flyash to lime ratio can be varied during full scale processing. Another factorial experiment will be performed to evaluate the addition of a "superplasticizer". The effects of curing the test cylinders at different temperatures will also be evaluated. Specimens from each of these three tests will be tested for all of the pertinent regulatory criteria listed above.

A petrographic analysis will be completed on specimens cured 28 days to determine the microscopic structure of the final CSS formulations.

Based on the Phase III results, a CSS formulation range for each type of pondcrete will be selected for full-scale testing. It may be possible that the same formulation will be acceptable for both forms of pondcrete.

A decision tree showing the CSS formulation screening and selection process for pondcrete is shown in Figure 1-4. A summary of the treatability study for pondcrete is presented in Table 1-4.

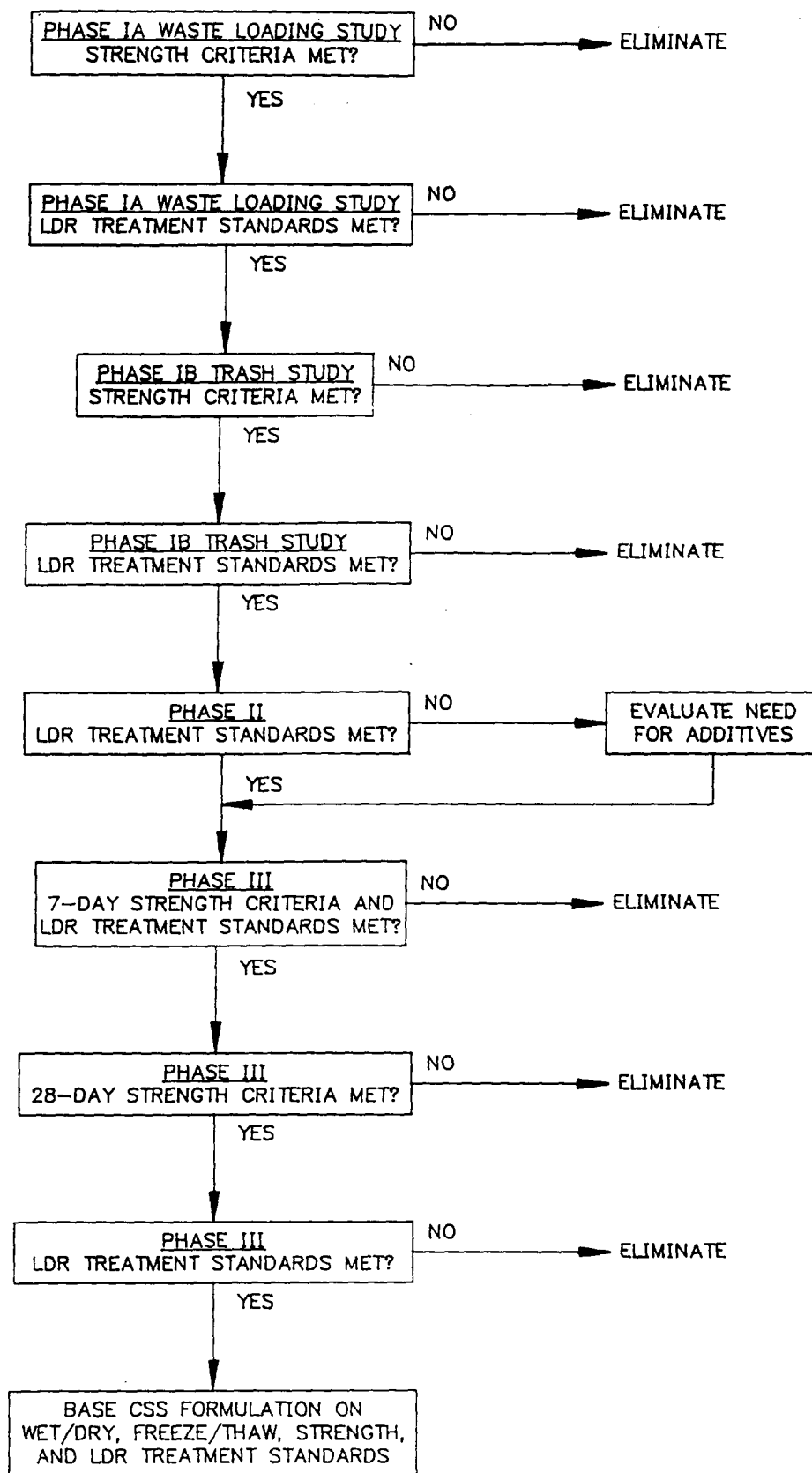


FIGURE 1-4

**CSS FORMULATION  
SCREENING AND SELECTION  
PROCESS FOR PONDCRETE  
ROCKY FLATS, GOLDEN, CO**



TABLE 1-4  
TREATABILITY STUDY SUMMARY FOR PONDCRETE  
ROCKY FLATS

Phase	CSS Binder	Additives	Mixes per Pondcrete Type	Cure Time	Tests	Tests per Pondcrete Type	Duration	Comments
IA	Methanol Study (1)	None	NA	NA	Total methanol	1	6 weeks	Duration: 6 weeks total. Goal: Determine whether methanol will leach above LDR treatment standard.
					ZHE TCLP - methanol	2	6 weeks	
					See Table 1-3	1	6 weeks	Goal: Determine baseline data to compare with characterization data.
	Baseline Analysis	None	NA	NA	Bulk density, moisture (gravimetric and Karl Fisher), specific gravity (total and as discrete particles)	1	4 weeks	Goal: Determine baseline for engineering (physical) parameters.
	Waste Loading Study	None	NA	NA	Total solids, TDS of supernatant, viscosity, specific gravity, bulk density	7	4 weeks	Duration: 7 weeks total. Goals: Determine upper limit of waste loading. Determine degree of dewatering required. Narrow selection of downstream process options.
					Density (mud balance), VG Fann testing	7	4 weeks	
					UCS, TCLP metals, accelerated wet/dry + UCS and freeze/thaw + UCS	7	7 weeks	
Dissolution Tests	None	None	NA	NA	Specific gravity and TDS of solution	12	2 weeks	Duration: 6 weeks total. Goals: Determine physical characteristics to evaluate handling options prior to mixing with pozzolans. Determine impact of trash on physical characteristics.
					Dry weight and specific gravity of undissolved portion	12	2 weeks	

TABLE 1-4  
TREATABILITY STUDY SUMMARY FOR PONDCRETE  
ROCKY FLATS  
PAGE 2

Phase		CSS Binder	Additives	Mixes per Pondcrete Type	Cure Time	Tests	Tests per Pondcrete Type	Duration	Comments	
IA Cont	Viscosity and Density Tests	None	None	NA	NA	Viscosity, Total solids and specific gravity of mixture	16	2 weeks	Continued	
						Viscosity of supernatant	5	2 weeks		
		Settling Tests	None	None	NA	NA	TDS, bulk density (terminal) and Total Solids	6	2 weeks	Duration: 6 weeks total. Goals: Determine physical characteristics to evaluate handling options prior to mixing with pozzolans. Determine impact of trash on physical characteristics.
		Rheology	None	None	NA	NA	Viscosity, specific gravity and Total Solids	6	2 weeks	
		Saturation TDS versus Temperature	None	None	None	NA	TDS, specific gravity	6	2 weeks	
1B	Dewatering Study	None	None	None	NA	By vendor	TBD	5 weeks	Goal: Evaluate dewatering processes.	
	Trash Study	None	None	10 (vary trash loading)	NA	Total solids, bulk density, viscosity, specific gravity, moisture (Karl Fisher)	10	3 weeks	Duration: 7 weeks total. Goal: Evaluate trash addition with respect to physical and chemical parameters.	
		Portland cement, flyash, and lime	None	30 (vary waste and trash loadings)	2 days	UCS, TCLP metals, accelerated wet/dry + UCS and freeze/thaw + UCS	30	6 weeks		
II CSS Formulation Development <sup>(2)</sup>		Portland cement, flyash, and lime	None	30 (vary water-to-pozzolan ratio and waste loadings)	Immediate	VG Fann, Density (mud balance)	30	3 weeks	Duration: 6 weeks total. Goal: Determine need for additives to reduce leaching of metals.	
					2 days	UCS, TCLP Metals, accelerated wet/dry + UCS and freeze/thaw + UCS	30			
		TBD <sup>(3)</sup>	TBD <sup>(3)</sup>	TBD	Immediate	VG Fann, Density (mud balance)	TBD	3 weeks		
					2 days	UCS, TCLP Metals	TBD			

TABLE 1-4  
TREATABILITY STUDY SUMMARY FOR PONDCRETE  
ROCKY FLATS  
PAGE 3

Phase	CSS Binder	Additives	Mixes per Pondcrete Type	Cure Time	Tests	Tests per Pondcrete Type	Duration	Comments
III	Regulatory Testing	TBD (3)	9 (vary water-to-pozzolan ratio and waste loading)	Immediate	VG Fann, Density (mud balance)	9	6 weeks	Duration: 10 weeks total. Goal: Determine whether CSS formulation will pass all regulatory criteria over proposed operating range.
				7 days	UCS, TCLP metals	9		
				28 days	UCS, TCLP metals PFLT, liquid/solid, wet/dry + UCS + TCLP metals (4), freeze/thaw + UCS + TCLP metals	9	10 weeks	
	Pozzolan Factorial	TBD (3)	5 (vary pozzolan ratios)	Immediate	VG Fann, Density (mud balance)	5	6 weeks	Duration: 10 weeks total. Goal: Evaluate variation of pozzolan ratio
				7 days	UCS, TCLP metals	5		
				28 days	UCS, TCLP metals, PFLT, liquid/solid, wet/dry (4), UCS + TCLP metals (4), freeze/thaw + UCS + TCLP metals	5	10 weeks	
	Curing Temperature	TBD (3)	6	Immediate	VG Fann, Density (mud balance)	6	6 weeks	Duration: 10 weeks total. Goal: Determine effect of curing temperature.
				7 days	UCS, TCLP metals	6		
				28 days	UCS, TCLP metals, PFLT, liquid/solid, wet/dry (4), UCS + TCLP metals (4), freeze/thaw + UCS + TCLP metals	6	10 weeks	
	Plasticizer	Super-plasticizer	3 (vary dosage)	Immediate	VG Fann, Density (mud balance)	3	6 weeks	Duration: 10 weeks total. Goal: Determine effect of superplasticizer addition.
				7 days	UCS, TCLP metals	3		
				28 days	UCS, TCLP metals, PFLT, liquid/solid, wet/dry (4), UCS + TCLP metals (4), freeze/thaw + UCS + TCLP metals	3	10 weeks	



TABLE 1-4  
TREATABILITY STUDY SUMMARY FOR PONDCRETE  
ROCKY FLATS  
PAGE 4

CSS -	Chemical Stabilization and Solidification
UCS -	Unconfined Compressive Strength
TCLP -	Toxicity Characteristic Leaching Procedure
PFLT -	Paint Filter Liquid Test
TBD -	To be determined
TDS -	Total dissolved solids
(1)	Triwalls in metal containers only
(2)	Triwalls only, unless concerns with presence and leaching of methanol
(3)	To be determined based on leaching of metals
(4)	TCLP metals analysis will be done on selected mid-point CSS formulations following durability cycling.

### 1.3.4 Pond 207C Slurry

#### 1.3.4.1 Assumptions

Pond 207C slurry will be screened to 10 mesh size. Material that does not pass 10 mesh will be ground to 10 mesh size. It is assumed that this can be done without adversely affecting the schedule.

The solids content of the slurry will be analyzed to determine/calculate dry solids. The amount of water and CSS formulation needed will be based on the dry solids of the slurry, after preliminary testing to determine acceptable CSS formulations.

Historically, there has been little or no success in developing completely successful CSS formulations for saltcrete because of the high salt content. High salt concentrations adversely affect cementation. The primary problem appears to be long-term durability as the salt within the cemented matrix crystallizes, grows, and expands, causing the solidified matrix to fail. In this treatability study, many CSS formulations, binders, and additives will be evaluated in an attempt to successfully solidify and stabilize Pond 207C slurry so that the solidified waste is durable and will not expand. Initial testing will evaluate both strength and wet/dry durability using various CSS formulations. In addition, a number of additives will be used in an attempt to overcome long-term durability problems. Even if a particular CSS formulation with additives solidifies the waste form, the long-term durability problem may not be completely solved. However, durability tests (wet/dry and freeze/thaw resistance) may provide an indication of how long the solidified waste remains stable if it needs to be stored on site for an extended period of time prior to shipment to the disposal facility.

The primary binders to be evaluated include portland cement only, lime plus flyash, and portland cement plus lime/flyash. The need for defoaming agents will also be evaluated. Based on literature reviews, foaming has been a problem when portland cement was mixed with salt solutions and brines. Foaming would add air to the solidified waste form. Foaming could result in air bubbles where salt crystals could grow and expand, thereby causing the solidified matrix to fail.

The treatability study schedule assumes the following:

- Accelerated curing of test specimens will be required in order to meet schedule constraints. Final testing to determine compliance with regulatory requirements and to verify long-term durability will be conducted on specimens that have cured for 28 days.
- Twelve batches (recipes) can be mixed per day (one shift).
- Two to four UCS tests can be conducted per hour.
- The TCLP metals analytical results will be available in 7 days, based on a maximum of 10 samples extracted per day.
- The results for full TCLP analysis (organics, if required, and inorganics) will be available in 6 weeks (maximum).
- Wet/dry and freeze/thaw durability testing will take 24 days to complete.

#### **1.3.4.2    Preliminary Testing**

The dry solids content of the pond slurry will be determined.

The slurry will be screened to 10 mesh size. Oversize material (greater than 10 mesh) will be ground to 10 mesh. This may be time-consuming; however, it may be necessary because of the presence of salt crystals from the bottom of Pond 207C.

Quick screening tests will be performed. These will involve mixing various binders (portland cement alone, lime plus flyash, and portland cement plus lime/flyash) and water with the pond slurry to obtain "workable" mixtures that, in the opinion of the laboratory technician, are neither too wet nor too dry. The initial starting point will be based on the experience of the laboratory technician, past experiences at Rocky Flats, and mixtures provided in literature for similarly characterized wastes. During these preliminary mixing tests, potential lime to flyash and portland cement to lime/flyash ratios will also be evaluated. In addition, if any of the binder/waste/water mixtures exhibits foaming, the need for a defoaming agent and the amount needed to reduce foaming problems will be evaluated.

The ratios of binder, waste, and water that are determined to be the best mixes based on this preliminary testing will be used as the starting points (center points) for the next phase of the treatability study.

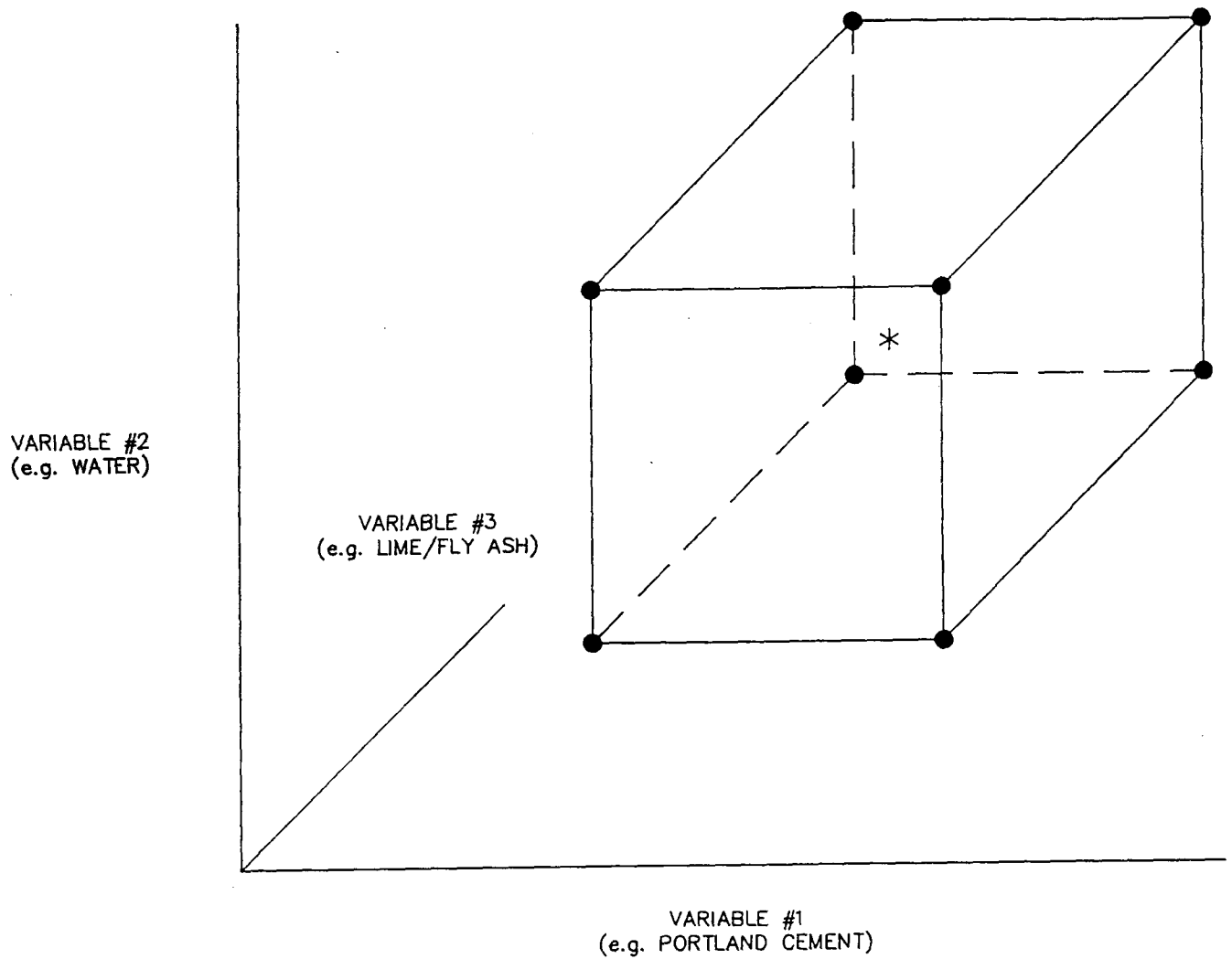
#### **1.3.4.3    Phase I**

This phase will be conducted in two steps. The first step of this phase involves experiments using three binder formulations: portland cement only, lime/flyash, and portland cement plus

lime/flyash. The experiment will bracket the center points determined in the preliminary testing. That is, the slurry concentration will be held constant, and the binder and water content will each be varied by a fixed percentage above and below the center point. The percent variation of the amount of binder(s) and water added will also be based on the preliminary testing. This means that for binders of portland cement only and lime/flyash three binder concentrations and three water concentrations will be evaluated. This will result in five recipes for each binder type (see Figure 1-2). For the binder that consists of portland cement plus lime/flyash, there will be nine recipes because the ratios of portland cement to lime/flyash will also be varied (see Figure 1-5). Therefore, for the first step of this phase of the treatability study for Pond 207C slurry, 19 recipes will be evaluated.

After the specimens for each formulation are mixed and placed into plastic test cylinders, they will be allowed to cure for 48 hours using the accelerated method. After curing, visual observations on free water content and the appearance of the specimens will be made. Formulations that pass the visual inspection will be subjected to UCS testing. Formulations that do not achieve sufficient strength will be eliminated from further consideration.

The second step of Phase I will basically repeat the step one testing. The recipes will be adjusted, as needed, to evaluate the range of acceptable mixes and to define the new center points, which would be the best anticipated recipe for each binder based on strength. This will result in the evaluation of an additional 19 recipes for Pond 207C slurry. After the specimens for each formulation are mixed and allowed to cure for 48 hours (accelerated method), they will be evaluated for strength.



#### LEGEND

- \* CENTER POINT CONCENTRATION
- CONCENTRATIONS  $\pm\%$   
AROUND CENTER POINT

**FIGURE 1-5**

**EXAMPLE OF THREE VARIABLES EVALUATED  
AT THREE CONCENTRATIONS  
ROCKY FLATS, GOLDEN, COLORADO  
NOT TO SCALE**



**HALLIBURTON NUS**  
Environmental Corporation

#### 1.3.4.4 Phase II

The binder formulations (using portland cement only; lime and flyash; and portland cement, lime, and flyash) that exhibit the best strength will each be tested in this phase. This phase of the treatability study for Pond 207C slurry will evaluate the addition of admixtures, such as latex, silica, and latex/silica combined. Three different concentrations of each of these admixtures will be tested for each of the selected binder formulations. This will result in 27 different formulations for evaluation. After the specimens for each formulation are mixed and allowed to cure for 48 hours (accelerated method), they will be evaluated for strength.

Formulations that pass the strength criteria will be leached using the TCLP method, and the extract will be analyzed for metals. Formulations that do not achieve the LDR treatment standards for the metals of concern will not be automatically eliminated from further consideration; however, the need for additives to reduce metal solubility may be evaluated in the next phase (Phase III).

Formulations that pass the strength criteria will be evaluated for wet/dry durability. The durability test specimens will also be evaluated for strength after the wet/dry durability test to determine whether a decrease in strength has occurred.

At this point in the treatability study for Pond 207C slurry, one binder formulation (portland cement only; lime and flyash; or portland cement, lime, and flyash) will be selected for further testing. All three admixtures (at one concentration each) will be retained for further testing, assuming that the use of a particular additive does not have a deleterious effect on strength or durability.

#### 1.3.4.5 Phase III

This phase of the treatability study will not be required if leaching of metals is not a problem, based on the TCLP results from Phase II. If leaching of metals is a problem, sulfide addition will be evaluated to reduce metal solubility. Sulfide will be used, rather than lime, because it is assumed that a sufficient hydroxide ion concentration will already be present in the binder formulations, from portland cement and/or lime. The sulfide concentration required will be based on stoichiometry to achieve minimum metals solubility. More than one sulfide concentration may be evaluated for each combination of the selected binder and each additive (latex, silica, and latex plus silica).

After the specimens for each formulation are mixed and allowed to cure for 48 hours (accelerated test), they will be evaluated for strength. Formulations that pass the strength criteria will be leached using the TCLP method, and the extract will be analyzed for metals. At this point it is assumed that at least one of the formulations (with silica added if necessary) will achieve the LDR treatment standards for the metals of concern.

#### 1.3.4.6 Phase IV

The selected binder formulations (portland cement only; lime and flyash; or portland cement, lime, and flyash) using the optimum concentration of each of the three additives (latex, silica, and latex plus silica) and using sulfide addition, if required, that exhibit the best strength and leachability characteristics will be tested in this phase. This phase of the treatability study will evaluate the use of an additional additive for strength (e.g., plastic fibers), long-term durability (i.e., wet/dry and freeze/thaw resistance), and regulatory compliance testing.



Six potential recipes will be evaluated for the selected binder (portland cement only; lime and flyash; or portland cement, lime, and flyash). These may include three recipes to evaluate admixtures (latex, silica, and latex plus silica) each with and without the additional strength additive. Duplicate specimens will be prepared for the proposed tests, except for wet/dry and freeze/thaw resistance where the tests use a control specimen.

During this phase, TCLP analysis will be conducted for the selected CSS formulation on a wet mix (i.e., less than 1 hour after the mixture is prepared). These results will be compared with TCLP analysis after a 28-day cure. This will be done to verify that TCLP metals concentrations will not fluctuate in an adverse manner from initial preparation to after a 28-day cure time. These results will be beneficial during actual remediation, thereby suggesting that samples collected during the initial pouring will indicate whether the billet will meet LDR requirements.

Additionally, two cylinders prepared with the selected CSS formulation will be cured for two days in a moist atmosphere, simulating expected conditions during remediation. These cylinders will be tested for UCS. The results will be compared to the 28-day UCS test results thereby providing a strength requirement after two days of curing. This data will provide a way to determine acceptability of a billet during remediation.

After mixing, the specimens will be allowed to cure for 7 days and evaluated for strength (UCS) and metals leachability (TCLP). For formulations where strength and leachability are acceptable, the specimens will be allowed to cure for 28 days total. After the 28-day cure, the formulations will be evaluated for strength, compliance with LDR treatment standards (TCLP and analysis for inorganics and organics of concern, if required), and durability

① (wet/dry and freeze/thaw resistance). Specimens that have been subjected to wet/dry and freeze/thaw tests will also be evaluated for strength to determine whether a decrease in strength has occurred.

A petrographic analysis will also be performed for each formulation to determine the microscopic structure of the solidified waste.

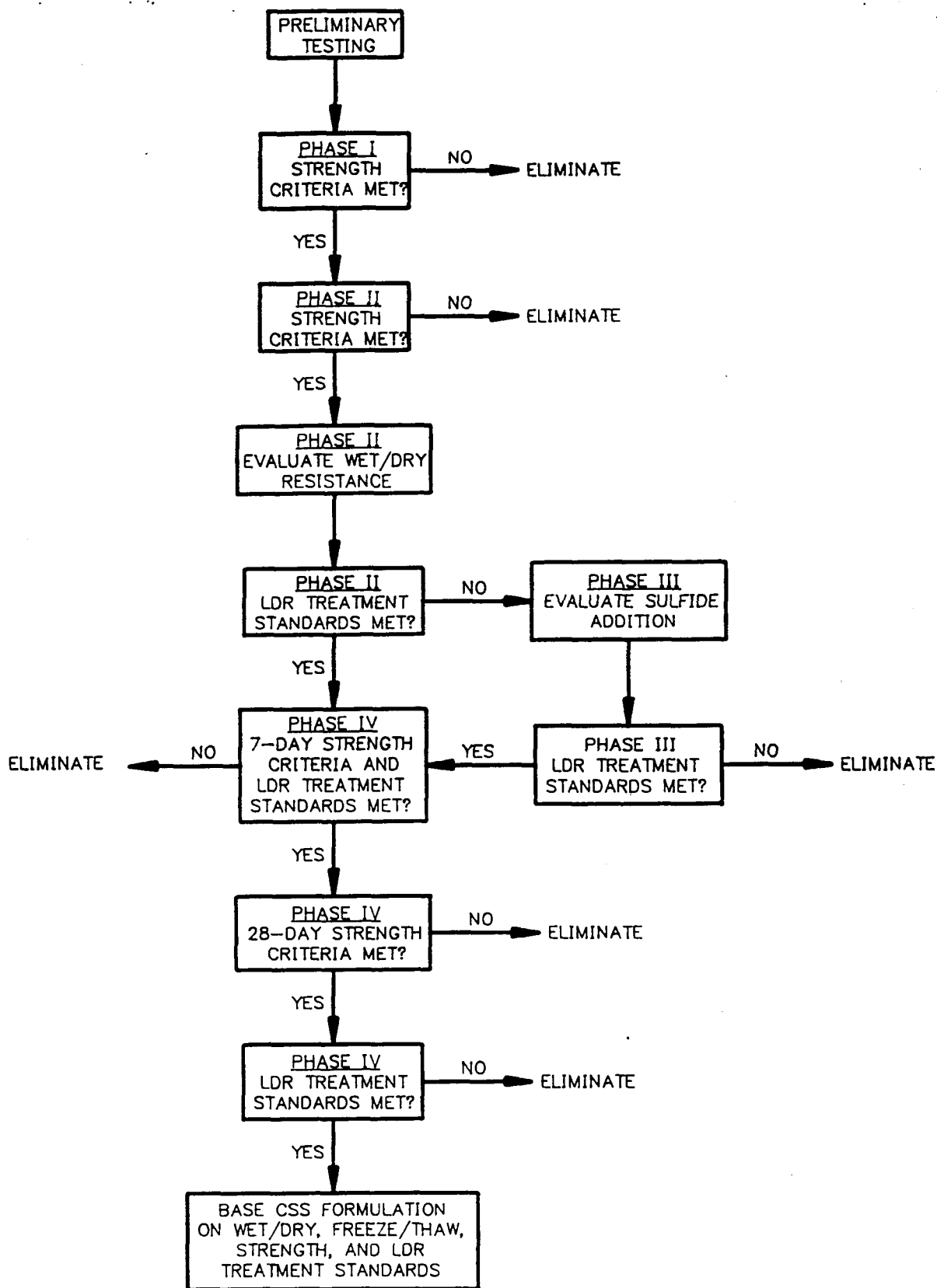
Based on the Phase IV results, a CSS formulation for Pond 207C slurry will be selected for use in full scale treatment.

A decision tree showing the CSS formulation screening and selecting process for Pond 207C slurry is presented in Figure 1-6. A summary of the treatability study for Pond 207C slurry is presented in Table 1-5.

#### **1.3.5      Saltcrete**

Saltcrete has recently been determined to consist of three separate waste forms, triwalls, half-crates, and triwalls in metal containers. The triwalls contain a waste loading of 50 to 55 percent (by weight) and half-crates primarily contain a waste loading of 33 to 35 percent (by weight). The metal containers store triwalls that have been damaged.

The treatability study for saltcrete will consist of three phases. A conceptual outline for the three phases is provided in Appendix H. Phase I will include physical tests and other testing necessary to determine major process options, thus allowing early selection of equipment. Phase I will consist of two sub-phases (IA and IB). Phase IA will consist of tests to determine the upper limit of waste loading and other engineering parameters such as viscosity, bulk density, specific gravity, etc. Phase IB will consist of dewatering studies and tests to evaluate the affect of inclusion



**CSS FORMULATION SCREENING  
AND SELECTION PROCESS  
POND 207C  
ROCKY FLATS, GOLDEN, CO**

**FIGURE 1-6**



**HALLIBURTON NUS**  
Environmental Corporation

TABLE 1-5  
TREATABILITY STUDY SUMMARY FOR POND 207C SLURRY  
ROCKY FLATS

Phase	CSS Binder	Additives	No. of Mixes	Cure Time	Tests	No. of Tests	Duration	Comments
Preliminary	None	None	NA	NA	Dry solids	1		Duration: 2 weeks total. Evaluate need for defoamer.
	None	None	NA	NA	Size to 10 mesh	1		
	Portland Cement	None	TBD	NA	Visual	TBD		
	Lime: flyash	None	TBD	NA	Visual	TBD		
	Portland Cement and Lime: flyash	None	TBD	NA	Visual	TBD		
Phase I Step 1	Portland Cement	None	5	2 days	UCS	5	1 week	Duration: 1 week total. Goal: Bracket center point of binder formulations based on strength and free liquid content.
	Lime: flyash	None	5	2 days	UCS	5	1 week	
	Portland Cement and Lime: flyash	None	9	2 days	UCS	9	1 week	
Phase I Step 2	Portland Cement	None	5	2 days	UCS	5	1 week	Duration: 1 week total. Goal: Define center point of binder mixtures based on strength and free liquid content.
	Lime: flyash	None	5	2 days	UCS	5	1 week	
	Portland Cement and Lime: flyash	None	9	2 days	UCS	9	1 week	
Phase II	Portland Cement	Silica flour	3	2 days	UCS, TCLP metals, Wet/dry + UCS	3		Duration: 5 weeks total. After Phase II testing completed, select one CSS binder for further evaluation. Goal: Define center point of binder + additive mixtures based on strength, leachability of metals and long term durability.
	Portland Cement	Latex	3	2 days	UCS, TCLP metals, Wet/dry + UCS	3		
	Portland Cement	Silica flour and latex	3	2 days	UCS, TCLP metals, Wet/dry + UCS	3		
	Lime: flyash	Silica flour	3	2 days	UCS TCLP metals, Wet/dry + UCS	3		
	Lime: flyash	Latex		2 days	UCS, TCLP metals, Wet/dry + UCS	3		
	Lime: flyash	Silica flour	3	2 days	UCS, TCLP metals, Wet/dry + UCS	3		
	Portland Cement and Lime: flyash	Latex	3	3 days	UCS, TCLP metals, Wet/dry + UCS	3		
	Portland Cement and Lime: flyash	Latex	3	2 days	UCS, TCLP metals, Wet/dry + UCS	3		
	Portland Cement and Lime: flyash	Silica flour and latex	3	2 days	UCS, TCLP metals, Wet/dry + UCS	3		

TABLE 1-5  
TREATABILITY STUDY SUMMARY FOR POND 207C SLURRY  
ROCKY FLATS  
PAGE 2 OF 3

Phase	CSS Binder	Additives	No. of Mixes	Cure Time	Tests	No. of Tests	Duration	Comments
Phase III	TBD and sulfide	Silica	1	2	UCS, TCLP metals	1	2 weeks	Duration: 2 weeks total.
	TBD and sulfide	Latex	1	2 days	UCS, TCLP metals	1	2 weeks	Goal: Define center point of binder + additive mixtures based on strength, leachability of metals and long term durability.
	TBD and sulfide	Silica flour and latex	1	2 days	UCS, TCLP metals	1	2 weeks	
Phase IV	TBD	Latex	1	None <sup>(1)</sup>	TCLP metals	1	1 week	Duration: 10+ weeks total.
				2 days <sup>(2)</sup>	UCS	2	1 week	Goal: Define mixture ratio at binder + additives to meet all LDR requirements and long term durability.
				7 days	UCS, TCLP metals	1	2 weeks	
				28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	
	TBD	Latex and plastic fibers	1	None <sup>(1)</sup>	TCLP metals	1	1 week	
				2 days <sup>(2)</sup>	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	
				28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	
	TBD	Silica flour	1	None <sup>(1)</sup>	TCLP metals	1	1 week	
				2 days <sup>(2)</sup>	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	
				28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	

TABLE 1-5  
TREATABILITY STUDY SUMMARY FOR POND 207C SLURRY  
ROCKY FLATS  
PAGE 3 OF 3

Phase	CSS Binder	Additives	No. of Mixes	Cure Time	Tests	No. of Tests	Duration	Comments
Phase IV (Cont.)	TBD	Silica flour and plastic fibers	1	None (1)	TCLP metals	1	1	
				2 days (2)	UCS	2	1 week	
				28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	
	TBD	Silica flour and latex	1	None (1)	TCLP metals	1	1 week	
				2 days (2)	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	
	TBD	Silica flour, latex and plastic fibers	1	28 days	UCS, PFLT, TCLP, wet/dry + UCS, freeze/thaw + UCS	1	10 weeks	
				None (1)	TCLP metals	1	1 week	
				2 days (2)	UCS	2	1 week	
				7 days	UCS, TCLP metals	1	2 weeks	

CSS - Chemical Stabilization and Solidification  
TBD - To Be Determined Based On Visual Observations Or Results From Previous Phase  
UCS - Unconfirmed Compressive Strength  
TCLP - Toxicity Characteristic Leaching Procedure  
PFLT - Paint Filter Liquid Test

(1) To be performed less than 1 hour after mixing.  
(2) Air dry curing.

of various amounts of trash. Phase II will consist of testing to screen various cement stabilization and solidification (CSS) formulas for key parameters, including any additives needed for compliance with land disposal restrictions (LDRs). Phase II will also include accelerated durability and UCS testing. Phase III will consist of testing to determine appropriate operating ranges for key process parameters and to demonstrate full regulatory compliance. Phase III will also include additional studies for miscellaneous concerns such as the affect of different curing temperatures and addition of a "superplasticizer". The results from preliminary phases may cause subsequent tests to be revised from that contained in this work plan.

The saltcrete testing will be performed using triwalls, triwalls in metal containers, and half-crates, unless otherwise noted.

#### **1.3.5.1 Assumptions**

Three types of saltcrete (from triwalls, triwalls in metal containers, and half-crates) will be subject to testing because the materials vary in consistency.

The solids content of the saltcrete will be analyzed to determine and calculate dry solids. The CSS formulation will be based on the dry solids of the saltcrete. The fluidity limit will be determined for saltcrete. This will dictate how much water must be added for the process stream.

The preliminary treatment process design assumes that saltcrete will be crushed, slurried to remove the oversize material, and then mechanically dewatered prior to mixing with the selected binder and water. Dewatering tests will be performed by vendors to determine the achievable sludge cake solids. CSS formulations for saltcrete

will be based on moisture content; therefore, necessary adjustments to CSS formulations and water to be added under full scale conditions can be based on these results.

Saltcrete samples will be ground/milled to 10 mesh screen size during field sampling so size reduction will not be needed during treatability study work conducted in the Pittsburgh Laboratory.

The treatability study schedule assumes the following:

- Accelerated curing of test specimens will be required in order to meet schedule constraints. Final testing to determine compliance with regulatory requirements and to verify long-term durability will be conducted on specimens that have cured for 28 days.
- Eight to twelve batches (recipes) can be mixed per day (one shift).
- Two to four UCS tests can be conducted per hour.
- The TCLP metals analytical results will be available in 7 days, based on a maximum of 10 samples extracted per day and low radioactivity of samples.
- The results for full TCLP analysis (inorganics and organics, if required) will be available in 6 weeks (maximum).
- Wet/dry and freeze/thaw long-term durability testing will take 24 days to complete (after specimens are cured).



#### 1.3.5.2 Phase IA

Phase IA consists of initial analytical testing and engineering studies.

##### Initial Analytical Testing of Feed Waste

Initial testing of the saltcrete waste forms includes a total dissolved solids (TDS) test and a baseline chemical analysis.

##### 1. Total Dissolved Solids (TDS) Test

Samples of each of the three saltcrete waste forms will be dissolved in excess deionized water at various dilutions. The liquid phase will then be filtered, and the filtrate will be analyzed for TDS. The filter cake will be analyzed for total solids and specific gravity. This will provide an initial baseline of the salt content of each of the waste forms.

##### 2. Baseline Analysis

The baseline analysis will determine baseline chemical data for comparison with characterization data. Representative samples for the three waste forms will be analyzed for the same parameters as the pondcrete waste forms (see Table 1-3).

##### Engineering Parameters

Representative samples of saltcrete in triwalls, triwalls in metal containers, and half-crates will be analyzed for bulk density, moisture content (gravimetric and Karl Fisher percent water), and specific gravity (total and as discrete particles). These analyses will form the baseline for engineering parameters.

## Engineering Studies

### 1. Waste Loading Study

The goal of the waste loading study is to determine the upper limit of the waste loading (% feed waste in the product slurry), which will determine the degree of dewatering, if any, that will be necessary (i.e., if 50 percent solids was the upper waste loading, then it would not be necessary to dewater the waste material to greater than 50 percent solids). The results of this study should narrow the selection of downstream process options. In addition, this testing will determine whether the addition of latex is beneficial and should be considered further during the CSS formulation development.

This study will consist of mixing the waste forms, at different solids concentrations, with the CSS formulation used for the solar ponds. The CSS formulation will consist of Type V portland cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.75, respectively. Batches will also be prepared using portland cement/flyash/hydrated lime and latex. Latex will be evaluated at concentrations of 0, 5, and 10 percent of the weight of the portland cement. Mixes will be conducted at solids loadings of 20, 30, 40, 50, 60, and 70 percent. The liquid phase, with the solids concentration at the saturation point, will also be solidified using the same CSS formulation. All mixes will be prepared at a water-to-pozzolan ratio of 0.42. A corresponding TDS value for each waste loading will also be determined. To assure uniform batches, enough material to mix all scheduled batches will be produced and stored in a sealed container until needed.

Analysis of the input (i.e., prior to mixing with pozzolans and latex) will include total solids, total dissolved solids for the

supernatant, viscosity, specific gravity, and bulk density.

Analysis of the output (i.e., after mixing but prior to curing) will include bulk density, VG Fann testing, and observation of the ability of the material to be pumped (this testing will be filmed).

After the specimens for each solids and latex loading are mixed and placed into plastic cylinders, they will be cured for 48 hours using the accelerated curing method. After curing, visual observation of the free water content and appearance of the specimens will be made. Formulations that pass the visual inspection will be subjected to UCS, TCLP metal and analysis, and accelerated durability testing (i.e., wet/dry and freeze/thaw resistance). Specimens that have been subjected to durability testing will also be evaluated for strength to determine whether a decrease in strength has occurred. Formulations that do not achieve sufficient strength or fail LDR requirements will be eliminated from further evaluation.

## 2. Dissolution Tests

The goal of these tests is to determine the extent of feed waste dissolution when mixed with water. This test consists of dissolving a known mass of sample in excess water and determining the specific gravity and TDS of the solution. The dry weight of the undissolved portion will also be determined.

Another test will consist of dissolving the sample in a small amount of water to make a saturated solution. The TDS and specific gravity of the supernatant will be measured.

Both of these tests will be conducted at room temperature and 100°F.

### 3. Viscosity and Density at Various Solids Contents

The goal of these tests is to determine how the solids content affects the viscosity and density of the feed wastes. Various mixtures of the waste and water will be prepared at concentrations of 5, 10, 20, 30, 40, 50, and 60 percent solids (or up to the plastic limit). The viscosity, specific gravity, and TDS will be measured for each mixture. The viscosity of the supernatant, up to the saturation point, will also be determined. The tests will be performed at room temperature and repeated at 100°F to reflect the temperature rise expected in the grinding circuit.

### 4. Settling Tests

Settling tests will be conducted on saltcrete samples ground to 10 mesh to determine settling rates and terminal densities. These tests will be performed using a saturated solution as the liquid phase and will be conducted at solids concentration of 5, 10, 20, 30, 40, 50, and 60 percent. The tests will be performed at room temperature and at 100°F.

### 5. Evaluation of Rheology of Slurries

For these tests, the viscosity of slurries will be measured at solids contents of 5, 10, 20, 30, 40, and 50 percent, of feed waste, with and without trash included in the slurry. This testing will be conducted in a saturated solution. Each slurry with trash will include 15 percent dry weight of trash (i.e., pallet, plastic sheeting, steel, etc.). The "steel" material will include steel banding, nails and artifacts from the ball mill. The trash comprises approximately 8 percent of the total weight of a billet. When calculated using dry weights, approximately 15 percent of the billet is considered to be trash. The specific

gravity of each slurry will be measured with and without trash. The viscosity of the slurry is needed to define pumping, settling, and other material handling parameter.

#### 6. Saturation TDS versus Temperature

The goal of these tests is to measure the degree of salt dissolution at various temperatures. This will be determined by collecting samples of the supernatant at different temperatures (i.e., room temperature, 50°F, and 100°F). The samples will be analyzed for TDS and specific gravity.

#### 1.3.5.3 Phase IB

This portion of Phase I will consist of evaluating dewatering processes and performing a trash study. The dewatering study will evaluate the selected process option that is believed to be the most practical to achieve the target solids loading determined in the waste loading study (Phase IA). The trash study will evaluate various loading of trash to determine whether there is any impact from different loadings.

#### 1. Dewatering Study

This testing will be evaluate various dewatering devices that are capable of achieving the solids content derived in the waste loading study. Vendors will be solicited to conduct the dewatering tests. Dewatering testing will include the appropriate quantities of trash. Further details on these tests will be developed at the completion of the waste loading study.

## 2. Trash Study

This study will evaluate trash addition with regard to chemical and physical parameters. Testing will be conducted at the TDS concentration determined from the waste loading study. Trash will be added at concentrations of 2, 5, 7.5, 10, 12.5, 15, 20, 50, 75, and 100 percent of the waste loading. The objective of these tests is to generate data to eliminate trash as a future variable for the CSS formulation development to be conducted during Phase II.

After the samples are mixed with various percentages of trash, they will be tested for percent solids, percent TDS, bulk density, viscosity, specific gravity, and percent water (Karl Fisher).

CSS testing will be conducted to determine the effect of trash on the stability of the solidified product and TCLP criteria. The CSS testing will be conducted at three waste loadings. One of the waste loadings will be that selected from the initial waste loading study. The others will bracket either side of the initial loading (e.g., plus or minus 10 percent solids). The water-to-pozzolan ratio will be 0.42. The CSS formulation will consist of Type V portland cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.75, respectively. After the specimens for each formulation are mixed and placed into plastic test cylinders, they will be cured for 48 hours using the accelerated method. After curing, visual observations of free water content and the appearance of the specimens will be made. Formulations that pass the visual inspection will be subjected to UCS, TCLP for metals, and accelerated durability testing (i.e., wet/dry and freeze/thaw resistance). Specimens that have been subjected to durability testing will also be evaluated for strength to determine whether a decrease in strength has occurred. Formulations that do not achieve sufficient strength or fail LDR requirements will be

eliminated form further evaluation.

#### **1.3.5.4 Phase II - CSS Formulation Development**

This phase will be conducted for triwalls, triwalls in metal containers, and half-crates. These studies will be conducted at the selected solids loading based on the waste loading study. For triwalls in metal containers, the water-to-pozzolan ratios will be varied and will include ratios of 0.34, 0.42, and 0.50. The CSS formulation will consist of type V portland cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.75, respectively. Latex addition will be evaluated only if it was determined to be necessary based on the Phase IA waste loading study. The testing for triwalls in metal containers will determine whether other additives are needed to improve the characteristics of the solidified product so that it will pass the TCLP testing criteria.

The product slurry produced from these tests will be subjected to viscosity (VG Fann) and density (mud balance) testing. The VG Fann testing will yield get strength characteristics from the product slurries produced. The mud balance testing will determine the densities of the product slurries. Together these tests will provide the necessary data to engineer the product slurry handling systems.

The half-crates and triwalls will be evaluated using higher water-to-pozzolan ratios. Treatment to reduce the leachability of the chemical constituents is not required because neither of these waste forms exceed andy LDR treatment standard. Using a higher water-to-pozzolan ratio will reduce the volume of the output. A water-to-pozzolan ratio of 1.0 will be evaluated to determine whether a stable waste can be produced. The result of the parameters will be the same as for triwalls in metal containers.

After the specimens for each formulation are mixed and placed into plastic test cylinders, they will be cured for 48 hours using the accelerated method and analyzed for TCLP metals. In addition, the specimens prepared from half-crates and triwalls will be subjected to accelerated durability testing followed by evaluation of strength to determine whether the higher water-to-pozzolan ratio had detrimental effects on wet/dry and freeze/thaw resistance. If the TCLP results pass the LDR treatment standards, then Phase II will be complete. If not, further testing will be required to evaluate various additives (to be determined) to reduce the leachability of the metal constituents.

Testing in this phase will be dependent on the results of the waste loading study. Upon completion of the waste loading study, additional information will be available to thoroughly scope the CSS formulation development.

#### **1.3.5.5 Phase III - Regulatory Compliance Testing**

Phase III will test the CSS formulation to determine whether it will pass all regulatory criteria over the proposed operating range. The proposed operating range for triwalls in metal containers will be at a water-to-pozzolan ration of 0.34 to 0.50, with 0.42 being the center point. The operating range for triwalls and half-crates will be determined in Phase II. these water-to-pozzolan ratios will be tested at  $\pm 10$  percentage points around the waste loading selected from the Phase IA testing.

After mixing the product slurries will be subjected to VG Fann and mud balance testing. The specimens will be allowed to cure for 7 days and evaluated for strength (UCS) and metals leachability (TCLP). For formulations where strength and leachability are acceptable, the specimens will be allowed to cure for 28 days



total. After the 28-day cure, the specimens will be evaluated for strength, metals leachability (TCLP) for compliance with LDR treatment standards, free liquid (Paint Filter Liquid Test and Solid/Liquid Test), and durability (i.e., wet/dry and freeze/thaw resistance). Specimens that have been subjected to durability testing will also be evaluated for UCS and TCLP metals testing to determine whether a decrease in strength or an increase in the leaching metals has occurred.

Several additional tests will be performed during Phase III. A factorial experiment that varies the ratio of pozzolans will be conducted so that the cement to flyash to lime ratio can be varied during full scale processing. Another factorial experiment will be performed to evaluate the addition of a "superplasticizer". The effects of curing the test cylinders at different temperatures will also be evaluated. Specimens from each of these three tests will be tested for all of the pertinent regulatory criteria listed above.

A petrographic analysis will be completed on specimens cured 28 days to determine the microscopic structure of the final CSS formulations.

Based on the Phase III results, a CSS formulation range for each type of saltcrete will be selected for full-scale testing. It may be possible that the same formulation will be acceptable for all three forms of saltcrete.

A decision tree showing the CSS formulation screening and selection process for saltcrete is shown in Figure 1-7. A summary of the treatability study for pondcrete is presented in Table 1-6.

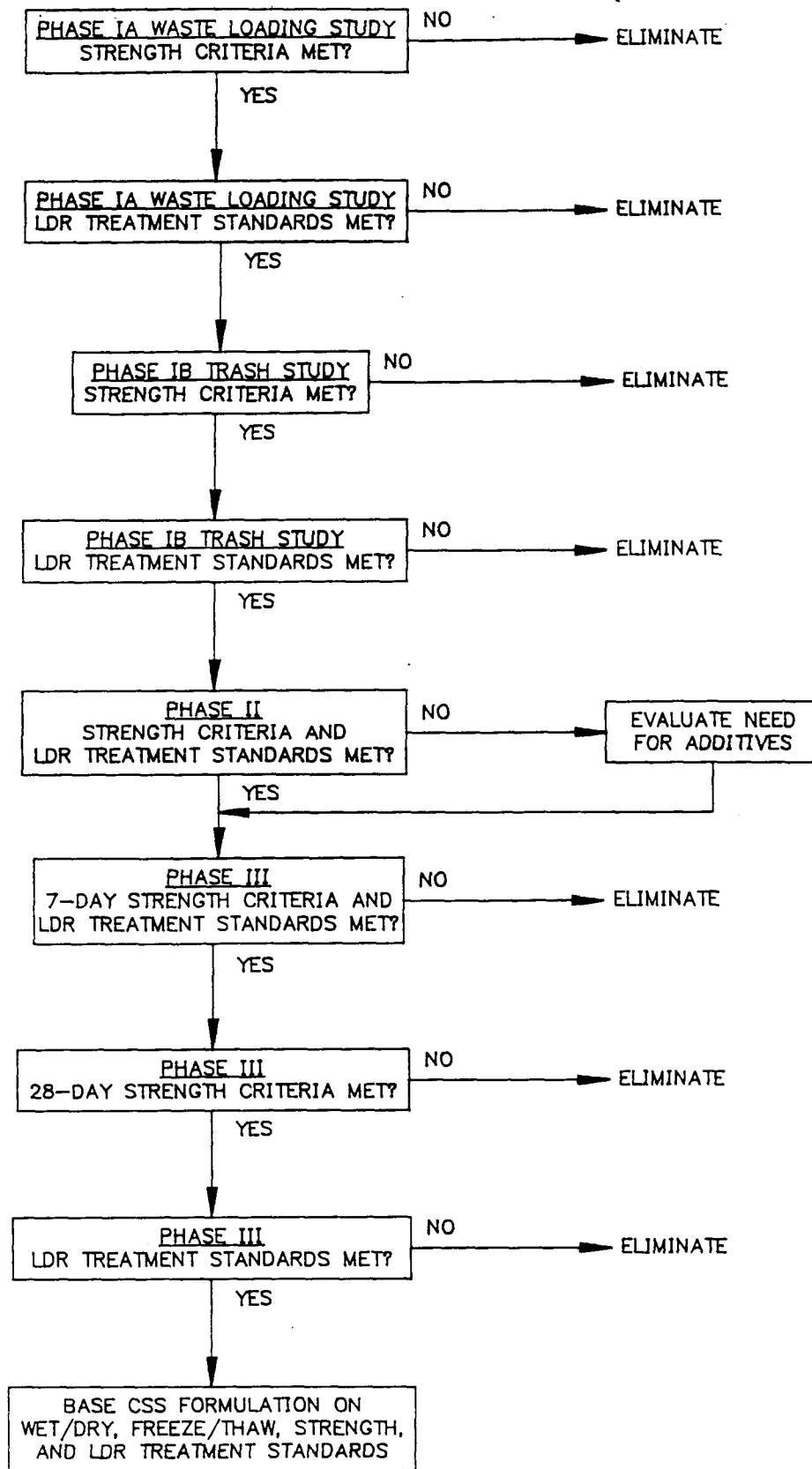


FIGURE 1-7

**CSS FORMULATION  
SCREENING AND SELECTION  
PROCESS FOR SALTCRETE  
ROCKY FLATS, GOLDEN, CO**



**HALLIBURTON NUS**  
*Environmental Corporation*

TABLE 1-6

TREATABILITY STUDY SUMMARY FOR SALTCRETE  
ROCKY FLATS

Phase	CSS Binder	Additives	Mixes per Saltcrete Type	Cure Time	Tests	Tests per Saltcrete Type	Duration	Comments
1A								
	TDS Tests	None	NA	NA	Liquid - TDS Solid - total solids and specific gravity	3	4 weeks	Goal: Provide initial baseline of salt content of each waste form.
	Baseline Analysis	None	NA	NA	See Table 1-3	1	6 weeks	Goal: Determine baseline data to compare with characterization data.
	Engineering Parameters	None	NA	NA	Bulk density, moisture (gravimetric and Karl Fisher), specific gravity (total and as discrete particles)	1	4 weeks	Goal: Determine baseline for engineering (physical) parameters.
	Waste Loading Study	None	NA	NA	Total solids, TDS of supernatant, specific viscosity, specific gravity, bulk density	21	4 weeks	Duration: 7 weeks total. Goals: Determine upper limit of waste loading. Determine degree of dewatering required. Narrow selection of downstream process options.
		Latex	21 (vary solids and latex loadings)	Immediate	Density (mud balance), VG Fann testing	21	4 weeks	
		Portland cement, flyash, and lime		2 days	UCS, TCLP metals, accelerated wet/dry + UCS and freeze/thaw + UCS	21	7 weeks	
Dissolution Tests	None	None	NA	NA	Specific gravity and TDS of solution	6	2 weeks	Duration: 6 weeks total. Goals: Determine physical characteristics to evaluate handling options prior to mixing with pozzolans. Determine impact of trash on physical characteristics.
					Dry weight and specific gravity of undissolved portion	6	2 weeks	

TABLE 1-6  
TREATABILITY STUDY SUMMARY FOR SALTCRETE  
ROCKY FLATS  
PAGE 2

Phase	CSS Binder	Additives	Mixes per Saltcrete Type	Cure Time	Tests	Tests per Saltcrete Type	Duration	Comments
1A Cont	None	None	NA	NA	Viscosity, Total solids, and specific gravity of mixture	16	2 weeks	Continued
					Viscosity of supernatant	5	2 weeks	
					TDS, bulk density (terminal), Total solids	6	2 weeks	
	None	None	NA	NA	Viscosity, specific gravity, Total Solids	6	2 weeks	Duration: 4 weeks total.
					TDS, specific gravity	6	2 weeks	
1B	None	None	NA	NA	By vendor	TBD	5 weeks	Goal: Evaluate dewatering processes.
	None	None	NA	NA	Total solids, TDS, bulk density, viscosity, specific gravity, % water (Karl Fisher)	10	3 weeks	Duration: 7 weeks total. Goal: Evaluate trash addition with respect to physical and chemical parameters.
					UCS, TCLP metals, accelerated wet/dry + UCS and freeze/thaw + UCS	30	6 weeks	

TABLE 1-6  
TREATABILITY STUDY SUMMARY FOR SALTCRETE  
ROCKY FLATS  
PAGE 3

Phase	CSS Binder	Additives	Mixes per Saltcrete Type	Cure Time	Tests	Tests per Saltcrete Type	Duration	Comments
II CSS Formulation Development	Portland cement, flyash, and lime	Latex <sup>(3)</sup>	30 (vary water-to-pozzolan ratio and waste loading)	Immediate	VG Fann, Density (mud balance)	30	3 weeks	Duration: 6 weeks total. Goal: Determine need for additives to reduce leaching of metals.
				2 days	UCS, TCLP, metals wet/dry + UCS <sup>(2)</sup> , freeze/thaw + UCS <sup>(2)</sup>	30		
	TBD <sup>(3)</sup>	TBS <sup>(3)</sup>	TBD	Immediate	VG Fann, Density (mud balance)	TBD	3 weeks	
				2 days	UCS, TCLP metals	TBD		
III	Regulatory Testing	Portland cement, flyash, and lime	9 (vary water-to-pozzolan ratio and waste loading)	Immediate	VG Fann, Density (mud balance)	9	10 weeks	Duration: 10 weeks total. Goal: Determine whether CSS formulation will pass all regulatory criteria over proposed operating range.
				7 days	UCS, TCLP metals	9		
				28 days	UCS, TCLP metals, PFLT, liquid/solid, wet/dry + UCS + TCLP metals <sup>(4)</sup> , freeze/thaw + UCS + TCLP metals <sup>(4)</sup>	9		
Pozzolan Factorial	Portland cement, flyash, and lime	TBD <sup>(3)</sup>	5 (vary pozzolan ratios)	Immediate	VG Fann, Density (mud balance)	5	10 weeks	Duration: 10 weeks total. Goal: Evaluate variation of pozzolan ratio.
				7 days	UCS, TCLP metals	5		
				28 days	UCS, TCLP metals, PFLT, liquid/solid, wet/dry + UCS + TCLP metals <sup>(4)</sup> , freeze/thaw + UCS + TCLP metals <sup>(4)</sup>	5	10 weeks	

TABLE 1-6  
TREATABILITY STUDY SUMMARY FOR SALTCRETE  
ROCKY FLATS  
PAGE 4

Phase	CSS Binder	Additives	Mixes per Saltcrete Type	Cure Time	Tests	Tests per Saltcrete Type	Duration	Comments
III Cont	Portland cement, flyash, and lime	TBD(3)	6 (vary temp.)	Immed-iate	VG Fann, Density (mud balance)	6	6 weeks	Duration: 6 weeks total. Goal: Determine effect of curing temperature.
				7 days	UCS, TCLP metals	6		
				28 days	UCS, TCLP metals, PFLT, liquid/solid, wet/dry(4) UCS + TCLP metals(4), freeze/thaw + UCS + TCLP metals	6	10 weeks	
Plasticizer	Portland cement, flyash, and lime	Super-plastic-izer	3 (vary dosage)	Immed-iate	VG Fann, Density (mud balance)	3	6 weeks	Duration: 10 weeks total. Goal: Determine effect of superplasticizer addition.
				7 days	UCS, TCLP metals	3		
				28 days	UCS, TCLP metals, PFLT, liquid/solid, wet/dry(4) UCS + TCLP metals(4), freeze/thaw + UCS + TCLP metals	3	10 weeks	

NA - Not Applicable

CSS - Chemical Stabilization and Solidification

UCS - Unconfined Compressive Strength

TCLP - Toxicity Characteristic Leaching Procedure

PFLT - Paint Filter Liquid Test

TBD - To be determined

TDS - Total dissolved solids

(1) Triwalls in metal containers only

(2) Triwalls only, unless concerns with presence and leaching of methanol

(3) To be determined based on leaching of metals

(4) TCLP metals analysis will be done on selected mid-point CSS formulations following durability cycling.

## **2.0 PROJECT ORGANIZATION AND MANAGEMENT**

### **2.1 Laboratory Access Control**

Laboratory access and security control are described in 3.4 "Facility Security" of Section QA-19, HALLIBURTON NUS Laboratory Services Group General Quality Assurance Plan.

Employees are required to wear a photo identification badge, All visitors must register at the front desk and must be escorted by a laboratory employee.

### **2.2 Project Organization**

The HALLIBURTON NUS Environmental Technologies Group (ETG) will be responsible for all activities conducted during the treatability study. Mr. Richard Ninesteel will serve as the Project Manager and will be responsible for the overall quality of the study including cost and schedule.

The treatability study will be conducted at the HALLIBURTON NUS Laboratory in Pittsburgh, Pennsylvania. Mr. Paul Frank will be the client contact and will be responsible for the quality of all work performed by the laboratory. Laboratory technicians will conduct all of the hands-on work for the study at the direction of ETG personnel.

### **2.3 Responsibility of Key Personnel**

Mr. Richard Ninesteel will be the Project Manager and will be responsible for the performance of the treatability study. Mr. Ninesteel will be assisted by Messrs. Mark Speranza, Tohomas Snare, and Robert Simcik. They will be assisting with various engineering tasks.

Mr. Paul Frank will be the client contact and will be responsible for all of the work conducted at the laboratory. The treatability study work will be conducted by Messrs. John Lehman and Donald Ahern. Other technicians will also assist in treatability study work as necessary.



### 3.0 QUALITY ASSURANCE OBJECTIVES

The waste treatability study objectives are summarized in Section 1.0. The data collection and quality assurance requirements described in this document are intended to provide data that are adequate in both number and quality to support completion of the treatability study.

#### 3.1 Data Quality Objectives

Data quality objectives (DQOs) are qualitative and/or quantitative statements regarding the quality of data needed to support the treatability study activities. In order to develop project-specific DQOs, the intended use of the data must be defined. This use must be balanced between data quality needs and time as well as cost constraints.

Specific analytical protocols are selected to meet the DQOs in the following ways:

- Compare data needs to the detection limits for available analytical methods.
- Select analytical methods to allow quantification of the analytes at levels sufficiently below the data needs to minimize the number of critical data points.
- Evaluate the maximum allowable variability in the data based on the data needs comparison.
- Develop project-specific acceptable variability based on the intended data use and method-specific precision and accuracy information.

Table 3-1 presents a summary of the proposed analysis program for the treatability study of Ponds 207A, 207B (Series), 207C and the clarifier. Table 3-2 presents a summary of the proposed analysis program for the pondcrete treatability study. A summary of the proposed analysis program for the saltcrete treatability study is presented in Table 3-3.

### 3.2 Precision, Accuracy, Representativeness, Completeness, and Comparability (PARCC) Goals

The quality of a data set is measured by certain characteristics of the data, namely the PARCC parameters. Some of the parameters are expressed quantitatively, while others are expressed qualitatively. The objectives of the waste characterization project and the intended use of the data define the PARCC goals.

Precision and accuracy characterize the amount of variability and bias inherent in a data set. Precision describes the reproducibility of measurements of the same parameter for a sample under the same or similar conditions. Precision is expressed as a range (the difference between two measurements of the same parameter) or as a relative percent difference (the range relative to the mean, expressed as a percent). Range and Relative Percent Difference (RPD) values are calculated as follows:

$$\text{Range} = \text{OR} - \text{DR}$$

and 
$$\text{RPD} = \frac{\text{OR} - \text{DR} \times 100\%}{1/2 (\text{OR} + \text{DR})}$$

where:

OR	=	original sample result
DR	=	duplicate sample result

TABLE 3-1

**SUMMARY OF TREATABILITY STUDY ANALYSIS PROGRAM  
PONDS 207A, 207B (Series), 207C AND CLARIFIER  
ROCKY FLATS**

207A Media	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates (A)	Matrix Spike/Matrix Duplicate	DOO Level (B)
Phase I	• Compression Test (UCS)	48 Hrs.	5	NA	ASTM D4289-83	0	NA	III
Phase II	• Compression Test (UCS) • TCLP (Cd, Cr, Pb, Ni, Ag, pH)	48 Hrs. 48 Hrs.	10 10	NA Per Method	ASTM D4289-83 SW 1311(3)	0 0	NA O/O	III III
Phase III	• Compression Test (UCS) • TCLP (Cd, Cr, Pb, Ni, Ag, pH) • Wet/Dry Test/Compression Test <sup>(1)</sup> • Freeze/Thaw Test/Compression Test <sup>(1)</sup> • Paint Filter Liquids Test • Petrographic Analysis • TCLP - Selected VOAs <sup>(2)</sup> - Selected Semivolatiles <sup>(2)</sup> - Selected Alcohols <sup>(2)</sup> - Cyanide (Total) - Cadmium - Chromium (Total) - Lead - Nickel - Silver - pH	7/28 Days 7 Days 28 Days 28 Days 28 Days 28 Days 28 Days	16/16 16 8/8 8/8 8 1 8 8 8 8 8 8 8 8 8 8	NA Per Method NA NA NA NA NA Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method	ASTM D4289-83 SW 1311(2) ASTM D559-82(4) ASTM D560-82(4) SW 9095 ASTM C856-77 SW 1311(3) SW 8240 SW 8270 ASTM D3695-82 ASTM D2036 SW 3050/6010 SW 3050/6010 SW 3050/6010 SW 3050/6010 SW 3050/6010 EPA 150.1	1/1 1 1 1 1 0 1 1 1 1 1 1 1 1 1 1 1	NA O/O NA NA NA NA NA 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 NA	III III III III III III III IV IV IV IV IV IV IV IV IV IV IV IV

TABLE 3-1  
SUMMARY OF TREATABILITY STUDY ANALYSIS PROGRAM  
PONDS 207A, 207B (Series), 207C AND CLARIFIER  
ROCKY FLATS  
PAGE TWO

207B Media	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DOO Level (B)
Phase I	• Compression Test (UCS)	48 Hrs.	5	NA	ASTM D4289-83	0	NA	III
Phase II	• Compression Test (UCS) • TCLP (Cd, Cr, Pb, Ni, Ag, pH)	48 Hrs. 48 Hrs.	10 10	NA Per Method	ASTM D4289-83 SW 1311 (3)	0 0	NA 0/0	III III
Phase III	• Compression Test (UCS) • TCLP (Cd, Cr, Pb, Ni, Ag, pH) • Wet/Dry Test/Compression Test (1) • Freeze/Thaw Test/Compression Test (1) • Paint Filter Liquids Test • Petrographic Analysis • TCLP - Selected VOAs (2) - Selected Semivolatiles (2) - Selected Alcohols (2) - Cyanide (Total) - Cadmium - Chromium (Total) - Lead - Nickel - Silver - pH	7/28 Days 7 Days 28 Days 28 Days 28 Days 28 Days 28 Days	16/16 16 8/8 8/8 8 1 8 8 8 8 8 8 8 8 8 8	NA Per Method NA NA NA NA NA Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method	ASTM D4289-83 SW 1311 (3) ASTM D559-82 (4) ASTM D560-82 (4) SW 9095 ASTM C856-77 SW 1311 (3) SW 8240 SW 8270 ASTM D3695-82 ASTM D2036 SW 3050/6010 SW 3050/6010 SW 3050/6010 SW 3050/6010 SW 3050/6010 SW 3050/6010 EPA 150.1	1/1 1 1 1 1 0 1 1 1 1 1 1 1 1 1 1 1	NA 0/0 NA NA NA NA NA 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 NA	III III III III III III III IV IV IV IV IV IV IV IV IV IV IV IV

TABLE 3-1  
SUMMARY OF TREATABILITY STUDY ANALYSIS PROGRAM  
PONDS 207A, 207B (Series), 207C AND CLARIFIER  
ROCKY FLATS  
PAGE THREE

207C Media	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DQO Level (B)
Phase I	• Compression Test (UCS)	48 Hrs.	38	NA	ASTM D4289-83	0	NA	III
Phase II	• Compression Test (UCS)	48 Hrs.	27	NA	ASTM D4289-83	0	NA	III
	• TCLP (Cd, Cr, Pb, Ni, Ag, pH)	48 Hrs.	27	Per Method	SW 1311(3)	0	0/0	III
	• Wet/Dry Test/Compression Test(1)	48 Hrs.	27/27	NA	ASTM D559-82	0	NA	III
Phase III	• Compression Test (UCS)	48 Hrs.	3	NA	ASTM D4289-83	0	0	III
	• TCLP (Cd Cr, Pb, Ni, Ag, pH)	48 Hrs.	3	Per Method	SW 1311(3)	0	0/0	III
Phase IV	• Compression Test (UCS)	7/28 Days	12/12	NA	ASTM D4289-83	1	NA	III
	• TCLP (Cd, Cr, Pb, Ni, Ag, pH)	7 Days	12	Per Method	SW1311(3)	1	0/0	III
	• Wet/Dry Test/Compression Test(1)	28 Days	12/12	NA	ASTM D559-82 (4)	1	NA	III
	• Freeze/Thaw Test/Compression Test(1)	28 Days	12/12	NA	ASTM D560-82 (4)	1	NA	III
	• Paint Filter Liquids Test	28 Days	12	NA	SW 9095	1	NA	III
	• Petrographic Analysis	28 Days	1	NA	ASTM C856-77	0	NA	III
	• TCLP	28 Days	12	Per Method	SW 1311(3)	1	1/1	IV
	- Selected VOAs (2)		12	Per Method	SW 8240	1	1/1	IV
	- Selected Semivolatiles (2)		12	Per Method	SW 8270	1	1/1	IV
	- Selected Alcohols (2)		12	Per Method	ASTM D3695-82	1	1/1	IV
	- Cyanide (Total)		12	Per Method	ASTM D2036	1	1/1	IV
	- Cadmium		12	Per Method	SW 3050/6010	1	1/1	IV
	- Chromium (Total)		12	Per Method	SW 3050/6010	1	1/1	IV
	- Lead		12	Per Method	SW 3050/6010	1	1/1	IV
	- Nickel		12	Per Method	SW 3050/6010	1	1/1	IV
	- Silver		12	Per Method	SW 3050/6010	1	1/1	IV
	- pH		12	Per Method	SW 3050/6010	1	1/1	IV
				NA	EPA 150.1	1	NA	IV

TABLE 3-1  
SUMMARY OF TREATABILITY STUDY ANALYSIS PROGRAM  
PONDS 207A, 207B (Series), 207C AND CLARIFIER  
ROCKY FLATS  
PAGE FOUR

Clarifier Media	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DOO Level (B)
Phase I	• Compression Test (UCS)	48 Hrs.	5	NA	ASTM D4289-83	0	NA	III
Phase II	• Compression Test (UCS) • TCLP (Cd, Cr, Pb, Ni, Ag, pH)	48 Hrs. 48 Hrs.	5 5	NA Per Method	ASTM D4289-83 SW 1311(3)	0 0	NA 0/0	III III
Phase III	• Compression Test (UCS) • TCLP (Cd, Cr, Pb, Ni, Ag, pH) • Wet/Dry Test/Compression Test <sup>(1)</sup> • Freeze/Thaw Test/Compression Test <sup>(1)</sup> • Paint Filter Liquids Test • Petrographic Analysis • TCLP - Selected VOAs <sup>(2)</sup> - Selected Semivolatiles <sup>(2)</sup> - Selected Alcohols <sup>(2)</sup> - Cyanide (Total) - Cadmium - Chromium (Total) - Lead - Nickel - Silver - pH	7/28 Days 7 Days 28 Days 28 Days 28 Days 28 Days 28 Days	16/16 16 8/8 8/8 8 1 8 8 8 8 8 8 8 8 8 8	NA Per Method NA NA NA NA NA Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method Per Method	ASTM D4289-83 SW 1311(3) ASTM D559-82(4) ASTM D560-82(4) SW 9095 ASTM C856-77 SW 1311(3) SW 8240 SW 8270 ASTM D3695-82 ASTM D2036 SW 3050/6010 SW 3050/6010 SW 3050/6010 SW 3050/6010 SW 3050/6010 EPA-150.1	1/1 1 1 1 1 0 1 1 1 1 1 1 1 1 1 1 1	NA 0/0 NA NA NA NA NA 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 1/1 NA	III III III III III III III IV IV IV IV IV IV IV IV IV IV

TABLE 3-1  
SUMMARY OF TREATABILITY STUDY ANALYSIS PROGRAM  
POMDS 207A, 207B (Series), 207C AND CLARIFIER  
ROCKY FLATS  
PAGE FIVE

- (A) Included for Land Disposal Restriction (LDR) analytes only.  
(B) Deliverables for DQO Level IV parameters will be as close to CLP requirements as possible. Deliverables for DQO Level III parameters will include signed and dated chain-of-custody forms, calculations, copies of analyst logbooks, and data summaries.

The following organics will only be analyzed if characterization analysis indicates that the compounds are present at concentrations such that they could leach at levels exceeding their respective standards. Otherwise, they will be deleted from the list of analytes.

- (1) Compression tests will be conducted after Freeze/Thaw and Wet Dry tests to determine if either test had detrimental effects on the strength of the cylinder. ASTM Method C39-86 will be followed for the compression test.

(2)		Selected Semivolatiles	Selected Alcohols
Selected VOAs	Tetrachloroethane	Cyclohexanone	n-Butyl Alcohol
	Trichloroethylene	Pyridine	Methanol
	Methylene Chloride	2-Nitropropane	Isobutanol
	1,1,1-Trichloroethane	1,2-Dichlorobenzene	2-Ethoxyethanol
	Carbon Tetrachloride		
	Chlorobenzene		
	1,1,2-Trichloro-1,2,2-Trifluoroethane		
	Trichlorofluoromethane		
	1,1,2-Trichloroethane		
	Xylene		
	Acetone		
	Ethyl Acetate		
	Ethylbenzene		
	Ethyl Ether		
	Methyl Isobutyl Ketone		
	Toluene		
	Methyl Ethyl Ketone		
	Carbon Disulfide		
	Benzene		

- (3) Extraction will be done as per SW 1311. Analysis of the listed compounds will be as per methods listed.  
(4) Modifications to these methods include: Cement to be set, uncompacted, in 2 in. dia. by 4 in. high polyethylene molds instead of compacted, metal molds. Curing time is to be Phase A specific instead of the method suggested 7-day time.

TABLE 3-2

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/ Matrix Duplicate	DOO Level (1)
IA	METHANOL STUDY							
	Total methanol	NA	1	Per Method	ASTM D3695-88	0	0	IV
	ZHE TCLP - methanol	NA	1	Per Method	SU 1311(2)	0	2	IV
	BASILINE ANALYSIS							
	TCLP - metals (As, Ba, Cd, Cr, Pb, Ni, Se, Ag, pH)	NA	2	Per Method	SU 1311(2)	0	Per Method	IV
	Total metals - Al, As, Sb, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Si, Na, Sr, Ti, Sn, V, Zn	NA	2	Per Method	SU 6010	0	Per Method	IV
	Total metals - Hg	NA	2	Per Method	SW 7471	0	Per Method	IV
	Alkalinity, carbonate(3)	NA	2	Per Method	EPA 310.1	0	0	III
	Alkalinity, phenolphthalein(3)	NA	2	Per Method	SM 403	0	0	III
	Alkalinity, MO(3)	NA	2	Per Method	SM 403	0	0	III
	Ammonia	NA	2	Per Method	EPA 350.2	0	0	III
	Chloride (3)	NA	2	Per Method	SW 9251	0	0	III
	Cyanide (total and amenable)	NA	2	Per Method	ASTM D2036-89	0	0	III
	Fluoride(3)	NA	2	Per Method	EPA 340.2	0	0	III
	Nitrate(3)	NA	2	Per Method	EPA 353.2	0	0	III
	pH	NA	2	Per Method	SW 9045	0	Per Method	IV
	Phosphorus (total)(3)	NA	2	Per Method	EPA 365.2	0	0	III
	Specific conductance(3)	NA	2	Per Method	EPA 120.1	0	0	III
	Sulfate(3)	NA	2	Per Method	SW 9036	0	0	III
	Sulfide(3)	NA	2	Per Method	SW 9030	0	0	III
	Total organic carbon (TOC)	NA	2	Per Method	Walkley-Black(2)	0	0	III
	ASTM leach - Cl, NO <sub>3</sub> , P, SO <sub>4</sub> , TDS	NA	2	Per Method	ASTM D3987-85(2)	0	0	III
	Americium (Am-241)	NA	2	Per Method	USDOE/EH-0053	0	0	III
	Plutonium (Pu-239/Pu-240)	NA	2	Per Method	AC-MM-2 0972	0	0	III
	ENGINEERING PARAMETERS							
	Bulk density	NA	2	NA	Agromony No. 9, Ch. 30	0	0	III
	Moisture (gravimetric)	NA	2	NA	ASTM D2216-90	0	0	III
	Moisture (Karl Fisher)	NA	2	NA	ASTM E203-75	0	0	III
	Specific gravity	NA	2	NA	ASTM D854-91	0	0	III
	Specific gravity (as discreet particles)	NA	2	NA	Air pycnometry (as per mfg.)	0	0	III



TABLE 3-2  
SUMMARY OF POND/CONCRETE TREATABILITY STUDY ANALYSIS PROGRAM  
ROCKY FLATS  
PAGE 2

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DOO Level (1)
1A cont.	WASTE LOADING STUDY							
	Total solids (waste feed)	NA	14	Per Method	EPA 160.3	0	0	III
	TDS (waste feed)	NA	14	Per Method	EPA 160.1	0	0	III
	Viscosity (waste feed)	NA	14	NA	ASTM D1084	0	0	III
	Specific gravity (waste feed)	NA	14	NA	ASTM D854-91	0	0	III
	Bulk density (waste feed)	NA	14	NA	Agronomy No. 9, Ch. 30	0	0	III
	Density (mud balance - CCS product)	Immediate	14	NA	Per Manufacturer (8)	0	0	III
	VG Fann testing (CCS product)	Immediate	14	NA	Per Manufacturer (9)	0	0	III
	Unconfined compressive strength (UCS)	2 days	14	NA	ASTM D4219-83	0	0	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	14	Per Method	SW 1311 (2)	0	Per Method	III
	Freeze/thaw test (4); UCS (5)	2 days	14	NA	ASTM D560-89 (6)	0	0	III
	Wet/dry test (4); UCS (5)	2 days	14	NA	ASTM D559-89 (6)	0	0	III
	DISSOLUTION TEST IN WATER							
	Specific gravity	NA	24	NA	ASTM D854-91	0	0	III
	Total dissolved solids	NA	24	Per Method	EPA 160.1	0	0	III
	Total solids	NA	24	Per Method	EPA 160.2	0	0	III
	VISCOSITY AND DENSITY AT DIFFERENT SOLIDS CONTENTS							
	Total solids	NA	32	Per Method	EPA 160.2	0	0	III
	Specific gravity	NA	32	NA	ASTM D854-91	0	0	III
	Viscosity	NA	42	NA	ASTM D1084	0	0	III
	SETTLING TESTS							
	Total dissolved solids	NA	12	Per Method	EPA 160.1	0	0	III
	Bulk density	NA	12	NA	Agronomy No. 9, Ch. 30	0	0	III
	Total solids	NA	12	Per Method	EPA 160.2	0	0	III
	RHEOLOGY OF SLURRIES							
	Total solids	NA	12	Per Method	EPA 160.2	0	0	III
	Specific gravity	NA	12	NA	ASTM D854-91	0	0	III
	Viscosity	NA	12	NA	ASTM D1084	0	0	III

TABLE 3-2  
SUMMARY OF POND/CONCRETE TREATABILITY STUDY ANALYSIS PROGRAM  
ROCKY FLATS  
PAGE 3

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DQO Level (1)
1A cont.	SATURATION TDS VERSUS TEMPERATURE							
	Specific gravity	NA	12	NA	ASTM D854-91	0	0	III
	Total dissolved solids	NA	12	Per Method	EPA 160.1	0	0	III
1B	DEWATERING STUDIES							
		NA	TBD	NA	NA	0	0	III
	TRASH STUDY							
	Bulk density	NA	20	NA	Agronomy No. 9, Ch. 30	0	0	III
	Moisture (Karl Fisher)	NA	20	NA	ASTM E203-75	0	0	III
	Specific gravity	NA	20	NA	ASTM D854-91	0	0	III
	Total solids	NA	20	Per Method	EPA 160.3	0	0	III
	Viscosity	NA	20	NA	ASTM D1084	0	0	III
	Unconfined compressive strength (UCS)	2 days	60	NA	ASTM D4219-83	0	0	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	60	Per Method	SW 1311(2)	0	Per Method	III
	Freeze/thaw test (4); UCS(5)	2 days	60	NA	ASTM D560-89(6)	0	0	III
	Wet/dry test (4); UCS(5)	2 days	60	NA	ASTM D559-89(6)	0	0	III
11	CSS FORMULATION DEVELOPMENT							
	VG Fann	Immediate	30	NA	Per Manufacturer(8)	0	0	III
	Mud balance	Immediate	30	NA	Per Manufacturer(9)	0	0	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	30	Per Method	SW 1311(2)	0	Per Method	III
	Freeze/thaw test (4); UCS(5)	2 days	30	NA	ASTM D560-89(6)	0	0	III
	Wet/dry test (4); UCS(5)	2 days	30	NA	ASTM D559-89(6)	0	0	III
	Unconfined compressive strength (UCS)	2 days	30	NA	ASTM D4219-83	0	0	III
As needed	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	TBD	Per Method	SW 1311(2)	0	Per Method	III

TABLE 3-2  
SUMMARY OF PONDCRETE TREATABILITY STUDY ANALYSIS PROGRAM  
ROCKY FLATS  
PAGE 4

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DQO Level (1)
III	REGULATORY COMPLIANCE TESTING							
	VG Fann	Immediate	64	NA	Per Manufacturer (8)	2	NA	III
	Mud balance	Immediate	64	NA	Per Manufacturer (9)	2	NA	III
	Unconfined compressive strength (UCS)	7/28 days	128	NA	ASTM D4219-83	2	NA	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	7/28 days	132	Per Method	SW 1311 (2)	2	Per Method	IV
	Paint Filter Liquids Test (PFLT)	28 days	64	NA	SW 9095	2	NA	III
	Solid/liquid test	28 days	64	NA	ASTM D4359-90	2	NA	III
	Freeze/thaw test; UCS <sup>(5)</sup> ; TCLP metals <sup>(5)</sup>	28 days	64	NA	ASTM D560-89 (6)	2	NA	III
	Wet/dry test; UCS <sup>(5)</sup> ; TCLP metals <sup>(5)</sup>	28 days	64	NA	ASTM D559-89 (6)	2	NA	III

(1) Deliverables for DQO Level IV parameters will be as close to CLP requirements as possible. Deliverables for DQO Level III parameters will include signed and dated chain-of-custody forms, calculations, copies of analysts notebooks, and data summaries.

(2) Extraction will be done as per method (SW 1311 or ASTM D3987-85). Analysis of listed parameters will be as per method listed elsewhere in table.

(3) Following dissolution in deionized water.

(4) Freeze/thaw and wet/dry methods (ASTM D560-89 and D559-89, respectively) are modified to shorten cycle time to 12 days.

(5) UCS tests (ASTM D4219-83) and TCLP metals (SW1311) will be conducted after durability tests to determine whether either tests had detrimental effects on strength or leachability of metals.

(6) Modifications to method include: cement to be uncompacted in 2-inch-diameter by 4-inch-high polyethylene molds instead of compacted in metal molds. Curing time is to be phase specific instead of method-suggested 7-day cure time.

(7) Included as Appendix G

(8) Included as Appendix E

(9) Included as Appendix F

ASTM American Society for Testing and Materials

EPA Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020

SW Test methods for Evaluating Solid Waste - Physical/Chemical methods, EPA SW-846, 3rd Edition

NA Not Applicable

TBD To Be Determined

TABLE 3-3  
SUMMARY OF SALICRETE TREATABILITY STUDY ANALYSIS PROGRAM  
ROCKY FLATS

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DOO Level (1)
1A	TDS TESTS							
	Specific gravity	NA	15	Per Method	ASTM D854-91	0	0	III
	Total dissolved solids	NA	15	Per Method	EPA 160.1	0	0	III
	Total solids	NA	15	Per Method	EPA 160.3	0	0	III
	BASELINE ANALYSIS							
	TCLP - metals (As, Ba, Cd, Cr, Pb, Ni, Se, Ag, pH)	NA	3	Per Method	SW 1311(2)	0	Per Method	IV
	Total metals - Al, As, Sb, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Si, Na, Sr, Tl, Sn, V, Zn	NA	3	Per Method	SW 6010	0	Per Method	IV
	Total metals - Hg (3)	NA	3	Per Method	SW 7471	0	Per Method	IV
	Alkalinity, carbonate (3)	NA	3	Per Method	EPA 310.1	0	0	III
	Alkalinity, phthalate (3)	NA	3	Per Method	SM 403	0	0	III
	Alkalinity, MO (3)	NA	3	Per Method	SM 403	0	0	III
	Ammonia (3)	NA	3	Per Method	EPA 350.2	0	0	III
	Chloride (3)	NA	3	Per Method	SW 9251	0	0	III
	Cyanide (total and amenable) (3)	NA	3	Per Method	ASTM D2036-89	0	Per Method	IV
	Fluoride (3)	NA	3	Per Method	EPA 340.2	0	0	III
	Nitrate (3)	NA	3	Per Method	EPA 353.2	0	0	III
	pH	NA	3	Per Method	SW 9045	0	0	III
	Phosphorus (total) (3)	NA	3	Per Method	EPA 365.2	0	0	III
	Specific conductance (3)	NA	3	Per Method	EPA 120.1	0	0	III
	Sulfate (3)	NA	3	Per Method	SW 9036	0	0	III
	Sulfide (3)	NA	3	Per Method	SW 9030	0	0	III
	Total organic carbon (TOC)	NA	3	Per Method	Walkley-Black	0	0	III
	ASTM leach - Cl, NO <sub>3</sub> , P, SO <sub>4</sub> , TDS	NA	3	Per Method	ASTM D3987-85(2)	0	0	III
	Americium (Am-241)	NA	3	Per Method	USDOE/EH-0053	0	0	III
	Plutonium (Pu-239/Pu-240)	NA	3	Per Method	AC-MM-2 0972	0	0	III
ENGINEERING PARAMETERS								
	Bulk density	NA	3	NA	Agronomy No. 9, Ch. 30	0	0	III
	Moisture (gravimetric)	NA	3	NA	ASTM D2216-90	0	0	III
	Moisture (Karl Fisher)	NA	3	NA	ASTM E203-75	0	0	III
	Specific gravity	NA	3	NA	ASTM D854-91	0	0	III
	Specific gravity (as discreet particles)	NA	3	NA	Air pycnometry (as per mfg.)	0	0	III

TABLE 3-3  
SUMMARY OF SALTRETE TREATABILITY STUDY ANALYSIS PROGRAM  
ROCKY FLATS  
PAGE 2

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DOO Level (f)
IA cont.	WASTE LOADING STUDY							
	Total solids (waste feed)	NA	21	Per Method	EPA 160.3	0	0	III
	TDS (waste feed)	NA	21	Per Method	EPA 160.1	0	0	III
	Viscosity (waste feed)	NA	21	NA	ASTM D1084	0	0	III
	Specific gravity (waste feed)	NA	21	NA	ASTM D854-91	0	0	III
	Bulk density (waste feed and CSS product)	NA	21	NA	Agronomy No. 9, Ch. 30	0	0	III
	Density (mud balance - CCS product)	Immediate	63	NA	Per Manufacture (8)	0	0	III
	VG Farm testing (CSS product)	Immediate	63	NA	Per Manufacture (9)	0	0	III
	Unconfined compressive strength (UCS)	2 days	63	NA	ASTM D4219-83	0	0	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	63	Per Method	SW 1311(2)	0	Per Method	III
	Freeze/thaw test (4); UCS (5)	2 days	63	NA	ASTM D560-89(6)	0	0	III
	Wet/dry test (4); UCS (5)	2 days	63	NA	ASTM D559-89(6)	0	0	III
	DISSOLUTION TEST IN WATER							
	Specific gravity	NA	18	NA	ASTM D854-91	0	0	III
	Total dissolved solids	NA	18	Per Method	EPA 160.1	0	0	III
	Total solids	NA	18	Per Method	EPA 160.2	0	0	III
	VISCOSITY AND DENSITY AT DIFFERENT SOLIDS CONTENTS							
	Total solids	NA	48	Per Method	EPA 160.2	0	0	III
	Specific gravity	NA	48	NA	ASTM D854-91	0	0	III
	Total dissolved solids	NA	48	Per Method	EPA 160.1	0	0	III
	Viscosity	NA	63	NA	ASTM D1084	0	0	III
	SETTLING TESTS							
	Total dissolved solids	NA	18	Per Method	EPA 160.1	0	0	III
	Bulk density	NA	18	NA	Agronomy No. 9, Ch. 30	0	0	III
	Total solids	NA	18	Per Method	EPA 160.2	0	0	III
	RHEOLOGY OF SLURRIES							
	Total solids	NA	18	Per Method	EPA 160.2	0	0	III
	Specific gravity	NA	18	NA	ASTM D854-91	0	0	III
	Viscosity	NA	18	NA	ASTM D1084	0	0	III

TABLE 3-3  
SUMMARY OF SALTCRETE TREATABILITY STUDY ANALYSIS PROGRAM  
ROCKY FLATS  
PAGE 3

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/ Matrix Duplicate	DOO Level (1)
1A cont.	SATURATION TDS VERSUS TEMPERATURE							
	Specific gravity	NA	18	NA	ASTM D854-91	0	0	III
	Total dissolved solids	NA	18	Per Method	EPA 160.1	0	0	III
1B	DEWATERING STUDIES							
		NA	TBD	NA	Per Manufacturer	0	0	III
	TRASH STUDY							
	Bulk density	NA	30	NA	Agronomy No. 9, Ch. 30	0	0	III
	Moisture (Karl Fisher)	NA	30	NA	ASTM E203-75	0	0	III
	Specific gravity	NA	30	NA	ASTM D854-91	0	0	III
	Total dissolved solids	NA	30	Per Method	EPA 160.1	0	0	III
	Total solids	NA	30	NA	EPA 160.3	0	0	III
	Viscosity	NA	30	Per Method	ASTM D1084	0	0	III
	Unconfined compressive strength (UCS)	2 days	90	NA	ASTM D4219-83	0	0	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	90	Per Method	SW 1311 (2)	0	Per Method	III
	Freeze/thaw test; UCS	2 days	90	NA	ASTM D560-89 (6)	0	0	III
	Wet/dry test; UCS	2 days	90	NA	ASTM D559-89 (6)	0	0	III
1I	CSS FORMULATION DEVELOPMENT							
	VG Fann	Immediate	90	NA	Per Manufacturer (8)	0	0	III
	Mud balance	Immediate	90	NA	Per Manufacturer (9)	0	0	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	90	Per Method	SW 1311 (2)	0	Per Method	III
	Freeze/thaw test; UCS	2 days	90	NA	ASTM D560-89 (6)	0	0	III
	Wet/dry test; UCS	2 days	90	NA	ASTM D559-89 (6)	0	0	III
	Unconfined compressive strength (UCS)	2 days	90	NA	ASTM D4219-83	0	0	III
As needed	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	2 days	TBD	Per Method	SW 1311 (2)	0	Per Method	III

TABLE 3-3  
SUMMARY OF SALTCRETE TREATABILITY STUDY ANALYSIS PROGRAM  
ROCKY FLATS  
PAGE 4

Phase	Analysis	Curing Time	Number of Samples	Target Detection Limit	Proposed Method	Laboratory Duplicates	Matrix Spike/Matrix Duplicate	DQO Level (1)
III	REGULATORY COMPLIANCE TESTING							
	VG Fann	Immediate	96	NA	Per Manufacturer (8)	3	NA	III
	Mud balance	Immediate	96	NA	Per Manufacturer (9)	3	NA	III
	Unconfined compressive strength (UCS)	7/28 days	192	NA	ASTM D4219-83	3	NA	III
	TCLP (As, Ba, Cd, Cr, Pb, Hg, Ni, Se, Ag)	7/28 days	96	Per Method	SW 1311 (2)	3	Per Method	IV
	Paint Filter Liquids Test (PFLT)	28 days	96	NA	SW 9095	3	NA	III
	Solid/liquid test	28 days	96	NA	ASTM D4359-90	3	NA	III
	Freeze/thaw test; UCS (5); TCLP metals (5)	28 days	96	NA	ASTM D560-89 (6)	3	NA	III
	Wet/dry test; UCS (5); TCLP metals (5)	28 days	96	NA	ASTM D559-89 (6)	3	NA	III

(1) Deliverables for DQO Level IV parameters will be as close to CLP requirements as possible. Deliverables for DQO Level III parameters will include signed and dated chain-of-custody forms, calculations, copies of analysts notebooks, and data summaries.

(2) Extraction will be done as per method (SW 1311 or ASTM D3987-85). Analysis of listed parameters will be as per method listed elsewhere in table.

(3) Following dissolution in deionized water.

(4) Freeze/thaw and wet/dry methods (ASTM D560-89 and D559-89, respectively) are modified to shorten cycle time to 12 days.

(5) UCS tests (ASTM D4219-83) and TCLP metals (SW1311) will be conducted after durability tests to determine whether either tests had detrimental effects on strength or leachability of metals.

(6) Modifications to method include: cement to be uncompacted in 2-inch-diameter by 4-inch-high polyethylene molds instead of compacted in metal molds. Curing time is to be phase specific instead of method-suggested 7-day cure time.

(7) Included as Appendix G

(8) Included as Appendix E

(9) Included as Appendix F

ASTM American Society of Testing and Materials

EPA Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020

SW Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, EPA SW-846, 3rd Edition

NA Not Applicable

TBD To Be Determined

The internal laboratory control limits for precision are three times the standard deviation of a series of RPD or range values. RPD values may be calculated for both laboratory and field duplicates, and can be compared to the control limits as a quality assurance check. Accuracy is the comparison between experimental and known or calculated values expressed as a percent recovery (%R). Percent recoveries are derived from analysis of standards spiked into deionized water (standard recovery) or into actual samples (matrix spike or surrogate spike recovery). Recovery is calculated as follows:

$$\%R = \frac{E}{T} \times 100\%$$

where:

E = Experimental result

T = True value or theoretical result

with

Theoretical result =  $\frac{(\text{Sample aliquot}) (\text{Sample conc.}) + (\text{Spike aliquot}) (\text{Spike Conc.})}{\text{Sample aliquot}}$

Control limits for accuracy are set at the mean plus or minus three times the standard deviation of a series of %R values. Organic %R values are set at the mean plus or minus two times the standard deviation.

Analytical precision and accuracy will be evaluated upon receipt of the laboratory data. Analytical precision will be measured as the relative standard deviation of the data from the laboratory (internal) duplicates. Analytical accuracy measures the bias as the percent recovery from matrix spike and surrogate spike samples. The requirements in Table 3-4 and Table 3-5 will be used for the organic analyses and the metal analyses.



**TABLE 3-4**

**SURROGATE SPIKE RECOVERY LIMITS**

**ROCKY FLATS FACILITY**

**SURROGATE SPIKE PERCENT RECOVERY LIMITS**

<b>Fraction</b>	<b>Surrogate Compounds</b>	<b>Water</b>	<b>Low/Medium Soil</b>
VOA	Toluene-d8	88-110	81-117
	4-Bromofluorobenzene	86-115	74-121
	1,2-Dichloroethane-d8	76-114	70-121

**TABLE 3-5****MATRIX SPIKE RECOVERY LIMITS  
ROCKY FLATS FACILITY****MATRIX SPIKE RECOVERY LIMITS**

<b>Fraction</b>	<b>Surrogate Compounds</b>	<b>Water</b>	<b>Low/Medium Soil</b>
VOA	1,1-Dichloroethene	61-145	59-172
	Trichloroethene	71-120	62-137
	Chlorobenzene	75-130	60-133
	Toluene	76-125	59-139
	Benzene	76-127	66-142
Metals	All metals of interest	75-125	75-125

No project resources will be expended to develop precision and accuracy data for method (field or analytical) validation except those commonly applied for collection of routine QA/QC data.

The "CLP like" deliverable and data validation will be performed on the DQO Level IV analysis only. DQO Level IV is considered legally defensible data and is needed to satisfy the LDR requirements. The analysis within DQO Level III is engineering study data and is to be utilized solely within the treatability study and will not be validated.

Validity of data (i.e., 95-percent confidence limit) with respect to its intended use will be assessed based on laboratory-supplied QA/QC data and protocols outlined in EPA's "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analysis 2/88" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses 7/88." In general, results that are rejected by the validation process will be disqualified from application for the intended use.

#### **4.0 LABORATORY SAMPLE CUSTODY**

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis. A brief summary for sample receipt, storage, and tracking is provided below. Further details are provided in Section QA-7 of the HALLIBURTON NUS Laboratory's General Quality Assurance Plan.

##### **4.1 Sample Receipt**

Samples arriving from the Rocky Flats facility will be treated as radioactive mixed waste. Upon sample receipt, radiation surveys shall be performed by the Radiation Safety Officer (RSO) or his designee. Containers will also be swipe-sampled and counted for gross alpha/beta activity to ensure that the outer drum has not been contaminated. If the sample radiation levels are greater than background-based instrument readings, they will be handled as radioactive materials and transferred to a controlled area.

##### **4.2 Sample Storage**

The sample custodian shall place the samples in the appropriate primary storage area. Samples are retained in the primary storage area until work is completed. Samples are retrieved and stored by Sample Custodians or Analysts.

##### **4.3 Laboratory Sample Tracking**

The HALLIBURTON NUS Pittsburgh Laboratory will maintain direct sample tracking showing the transfer of samples from secure, locked sample storage to the secure laboratory area and back to locked storage. To maintain chain-of-custody within the laboratory,

samples, sample extractions, and sample digestions are stored in locked areas when not in use for sample preparation or analysis. Keys to the locked storage areas are controlled by the Sample Custodian or designated laboratory personnel.

## **5.0 CALIBRATION PROCEDURES AND FREQUENCY**

Instruments used in the laboratory will be calibrated according to the procedures listed in the HALLIBURTON NUS Laboratory General Quality Assurance Plan, as summarized below:

### **5.1 Volatile Organics**

Calibration of the GC/MS instruments will take place once the instrument tune requirements of Section 7 of the method are met. Calibration of the GC/MS instruments will be done for all TCLP volatile compounds as per Section 2.0 of Appendix C of the laboratory's General Quality Assurance Plan. Ethyl acetate, ethyl ether, and 1,1,2-trichloro-1,2,2-trifluoroethane will be calibrated using a 1-point initial calibration.

### **5.2 Alcohols**

Calibration of the GC instruments will be done for the alcohols listed in Table 6-1 as per Section 2.0 of Appendix C of the laboratory's General Quality Assurance Plan except that a 3-point initial calibration will be performed.

### **5.3 Metals**

Calibration of the instruments used for the metals analyses will be done as per Section 1.0 of Appendix D of the laboratory's General Quality Assurance Plan.

### **5.4 Cyanide, pH, and Other Analytes**

These parameters will be calibrated according to Appendix E of the laboratory's General Quality Assurance Plan.

## **6.0 TREATABILITY STUDY PROCEDURES**

### **6.1 Analytical Procedures**

The analytical procedures to be used are summarized in Table 6-1.

### **6.2 Bench-Scale Design Procedures**

#### **6.2.1 Accelerated Curing Procedure**

The procedure will be as per ASTM C684-89 "Making, Accelerated Curing, and Testing of Concrete Compression Test Specimens" with the following modifications:

Molds - Single use, JATCO polyethylene 2-in. dia. by 4-in. high molds to be used. The top of the specimen is to be leveled and covered with paraffin then capped to prevent specimen loss during curing.

Curing time - 48 hrs.  $\pm$  30 min. shall be the duration of the curing time using Procedure A - Warm Water Method.  
- Slump and air content measurements will not be required.

#### **6.2.2 Mixing Procedure**

The particular wastes to be treated in the solidification study are:

- 207A Sludge
- 207B Sludge
- Clarifier Sludge
- 207C Slurry
- Pondcrete
- Saltcrete

TABLE 6-1

**ANALYTICAL SUMMARY  
TREATABILITY STUDY  
ROCKY FLATS**

ANALYSIS	METHOD	
	SOLID	LIQUID/EXTRACT
<b>PHYSICAL PARAMETERS</b>		
Accelerated curing	ASTM C684-89 <sup>(1)</sup>	---
Bulk density	Agronomy No. 9, Ch. 30	---
Freeze/thaw resistance	ASTM D560-89 <sup>(2)</sup>	---
Moisture (gravimetric)	ASTM D2216-90	---
Percent Water (Karl Fisher)	ASTM E203-75	---
Paint Filter Liquid Test	SW 9095	---
Solid/Liquid Test	ASTM D4359-90	---
Specific gravity	ASTM D854-91	---
Specific gravity (as discrete particles)	Air pycnometry (per mfg. suggested method) <sup>(4)</sup>	---
Unconfined compressive strength (UCS)	ASTM D4219-83	---
Wet/dry resistance	ASTM D559-89 <sup>(2)</sup>	---
VG Fann Viscometer	Per Manufacturer <sup>(5)</sup>	---
Mud balance	Per Manufacturer <sup>(6)</sup>	---
<b>LEACHING PROCEDURES</b>		
TCLP Leach	SW 1311	---
ASTM Leach	ASTM D3987-85	---
<b>ORGANICS</b>		
Methanol	ASTM D3695-88	ASTM D3695-88
<b>METALS</b>		
Aluminum	SW 3050/6010	---
Arsenic	SW 3050/6010	SW 3010/6010
Antimony	SW 3050/6010	---
Barium	SW 3050/6010	SW 3010/6010
Beryllium	SW 3050/6010	---
Boron	SW 3050/6010	---
Cadmium	SW 3050/6010	SW 3010/6010
Calcium	SW 3050/6010	---
Chromium, total	SW 3050/6010	SW 3010/6010
Cobalt	SW 3050/6010	---
Copper	SW 3050/6010	---
Iron	SW 3050/6010	---
Lead	SW 3050/6010	SW 3010/6010
Magnesium	SW 3050/6010	---
Manganese	SW 3050/6010	---
Mercury	SW 3050/7470	SW 3010/7470
Nickel	SW 3050/6010	SW 3010/6010
Potassium	SW 3050/6010	---
Selenium	SW 3050/6010	SW 3010/6010
Silicon	SW 3050/6010	---



TABLE 6-1  
ANALYTICAL SUMMARY  
TREATABILITY STUDY  
ROCKY FLATS  
PAGE 2

ANALYSIS	METHOD	
	SOLID	LIQUID/EXTRACT
METALS (CONT.)		
Silver	SW 3050/6010	SW 3010/6010
Sodium	SW 3050/6010	---
Strontium	SW 3050/6010	---
Thallium	SW 3050/6010	---
Tin	SW 3050/6010	---
Vanadium	SW 3050/6010	---
Zinc	SW 3050/6010	---
RADIONUCLIDES		
Americium (Am-241)	USDOE/EH-0053, August 1987 (gamma spec)	---
Plutonium (Pu-239/Pu-240)	AC-MM-2, 1972, Oak Ridge (Draft)	---
OTHERS		
Alkalinity, carbonate	EPA 310.1 <sup>(3)</sup>	---
Alkalinity, MO	SM 403 <sup>(3)</sup>	---
Alkalinity, phenolphthalein	SM 403 <sup>(3)</sup>	---
Ammonia	EPA 350.2	---
Chloride	SW 9251 <sup>(3)</sup>	---
Cyanide (total and amenable)	ASTM D2036-89	---
Fluoride	EPA 340.2 <sup>(3)</sup>	---
Nitrate	EPA 353.2 <sup>(3)</sup>	---
pH	SW 9045	SW 9045
Phosphorus, total	EPA 365.2 <sup>(3)</sup>	---
Specific conductance	EPA 120.1 <sup>(3)</sup>	---
Sulfate	SW 9096 <sup>(3)</sup>	---
Sulfide	SW 9030 <sup>(3)</sup>	---
Total dissolved solids	EPA 160.1	---
Total organic carbon	Walkley-Black	---
Total solids	EPA 160.3	---
Viscosity	---	ASTM D1084

**TABLE 6-1  
ANALYTICAL SUMMARY  
TREATABILITY STUDY  
ROCKY FLATS  
PAGE 3**

SW SW-846 Test Methods for Evaluation of Solid Waste, Physical/Chemical Methods, 3rd Edition.

EPA Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020.

ASTM American Society for Testing and Materials.

(1) Modifications to these methods include:

Stabilized waste to be set, uncompacted, in 2 in. dia. by 4 in. high polyethylene molds instead of compacted in metal molds.

Curing time is to be phase specific instead of the 7-day time suggested by the method.

(2) Modification to this method includes 48-hour cure rather than 24 hours

(3) Following dissolution in deionized water.

(4) Procedure included as Appendix G.

(5) Procedure included as Appendix E.

(6) Procedure included as Appendix F.

#### **6.2.2.1 Preparation**

Representative aliquots of the waste media to be solidified will be taken from the sample storage drums and, when specified, passed through the 10 mesh sieve to determine whether further grinding will be necessary.

#### **6.2.2.2 Weighing**

The waste and ingredients of the particular batch will be weighed on an analytical balance to the nearest 0.01g and the weight recorded.

#### **6.2.2.3 Mixing**

Ingredients for the particular batch are to be mixed using the Hobart mixer for 5 minutes. The speed of the mixing will be initially determined by the technician and will be held constant for all batches. Cylinders shall be filled completely and leveled off. After the cylinders are leveled off, paraffin will be used as well as capping to ensure a seal.

#### **6.2.2.4 Cleanup**

Equipment must be decontaminated (cleaned) before the next batch is mixed.

#### **6.2.3 Jar Test**

The Jar Test will provide the identity and concentration of the coagulant/polymer to effectively flocculate the sludge mixtures. Waste to be analyzed for this test are:

- 207A Sludge + Pond Water
- 207B Sludge + Pond Water
- 207A/B Sludge + Pond Water

Equipment and procedure will follow "Jar Tests;" pages 36-39, Water and Wastewater Technology by Mark J. Hammer. A copy of this procedure is provided in Appendix A.

#### **6.2.4 Bulk Settling Rate Test**

The Bulk Settling Rate Test will provide the data necessary to determine the size of clarifier needed. Wastes to be analyzed for this test are:

- 207A Sludge + Pond Water with/without coagulant/polymer
- 207B Sludge + Pond Water with/without coagulant/polymer
- 207A/B Sludge + Pond Water with/without coagulant/polymer

The sludge mixtures are to be sieved to less than 325 mesh with the coagulant/polymer concentration determined by the Jar Test. A copy of this procedure is provided in Appendix B.

#### **6.2.5 Disaggregation Test**

The Disaggregation Test will determine the effect of pond water on the structural integrity of the previously solidified wastes. This will provide useful information for milling considerations within the process. Wastes to be analyzed for this test are:

- Pondcrete
- Saltcrete

A copy of the procedure is provided in Appendix C.

#### **6.2.6 Pressure Filter Test**

This test is to be performed by Larox personnel at the HALLIBURTON NUS Laboratory using the Larox Laboratory Filter Labox 25. The purpose of this test is to determine the maximum percent solids achievable through pressure filtration of the particular waste. Wastes to be analyzed for this test are:

- Pondcrete
- Saltcrete

Pondcrete and saltcrete will be used on an "as-received" basis (<10 mesh). The percent solids of these mixtures will be determined from the preliminary design of the process. A copy of the procedure to be used is provided in Appendix D.

#### **6.2.7 Viscosity Test**

Viscosity testing will be preformed using a Fann V-G Viscometer. This equipment is capable of providing the plastic viscosity, yield point, and gel strength of the product slurry. Consistency curves and thixotropic studies can be preformed from the data acquired. Wastes to be analyzed for this test are:

- 207A/B with Pozzolons
- 207C with Pozzolons
- 207C and Clarifier with Pozzolons
- Pondcrete with Pozzolons
- Saltcrete with Pozzolons

The procedure will follow manufacturers suggested operating instructions as provided in Appendix E.

#### **6.2.8 Mud Balance Test**

The mud balance testing uses the TRU-WATE cup for measuring the absolute density of cement slurries. This modified procedure enables the product slurries to be placed under pressure to eliminate the air entrained in the slurries to get an accurate density of the material. Waste to be analyzed for this test are:

- 207A/B with Pozzolons
- 207C with Pozzolons
- 207C and Clarifier with Pozzolons
- Pondcrete with Pozzolons
- Saltcrete with Pozzolons

A step-by-step procedure for the operating of the TRU-WATE cup is provided in Appendix F.

#### **6.2.9 Pycnometer Testing**

The bulk densities of the product slurries will be determined using a multipycnometer. The equipment used is a Quantachrome Corporation's multipycnometer model MVP-1. Waste to be analyzed for this test are:

- 207A/B with Pozzolons
- 207C and Pozzolons
- 207C/Clarifier and Pozzolons
- Pondcrete with Pozzolons
- Saltcrete with Pozzolons

The procedure for determining the bulk density by multipycnometer is as described in the "Onsite Lab Operating Instructions, Deliverable #316, Section RF-10" and provided in Appendix G.

#### **6.2.10 Disposal of Laboratory Wastes**

The amount of radioactive material permitted in the laboratory at any one given time is strictly regulated. It is of vital importance that all residual material, unused sample, and all solid and liquid wastes produced in these studies be removed from the laboratory before additional feed wastes are accepted for study.

The details of the disposal and/or removal of the wastes generated in the Treatability Study is discussed in the letters provided in Appendix I.

## **7.0 INTERNAL QUALITY CONTROL CHECKS**

Quality Control checks to be implemented in the laboratory are described in this section. Laboratory analyses will be conducted in accordance with the appropriate analytical methods (See Table 6-1). Internal laboratory quality control checks may include surrogate and matrix spike addition and analysis and reagent blank generation and analysis. Internal laboratory quality control checks are described below.

### **7.1 Volatile Organics**

The quality control procedures routinely employed in gas chromatography/mass spectrometry (GC/MS) analyses of water and soil/sediment for Volatile Organics Analysis (VOAs) are presented below:

#### **7.1.1 Mass Spectrometer Calibration and Instrument and Column Performance Evaluation**

The mass assignment and resolution of the mass spectrometer is calibrated using perfluorotributylamine (FC-43). After that, the system performance is evaluated daily by injecting 50 ng of bromofluorobenzene (BFB) for VOAs. The ion abundance criteria specified in the method must be met before any analyses are attempted. If difficulty is encountered in meeting the criteria for BFB, the mass spectrometer is turned and re-evaluated until the requirements for BFB are met.

#### **7.1.2 Standardization**

The GC/MS is calibrated initially with five standards covering the working range of the instrument.



Each day that analyses are performed, the instrument is standardized against a standard solution containing the compounds of interest. A Response Factor (RF) is determined by the Internal Standard (IS) method each time the standardization is performed. Internal standards, which are usually deuterated compounds, are added to all standards as well as to all blanks and samples before injection into the GC/MS system. For VOAs, three internal standards are added. Internal standard areas for each sample are monitored to detect changes in instrument conditions. The standardization response factor is calculated as follows:

$$RF = \frac{A_S C_{IS}}{A_{IS} C_S}$$

Where:

- $A_S$  = integrated area of the characteristic ion for the compound in the standard.
- $A_{IS}$  = integrated area of the characteristic ion for the internal standard.
- $C_{IS}$  = amount (mg) of the internal standard.
- $C_S$  = amount (mg) of the compound in the standard.

A constant amount of internal standard is added to all samples, and the concentration ( $C_o$ ) of the compound in the sample is calculated using the following equation:

$$C_o \text{ (ug/l)} = \frac{(A_S) (C_{IS})}{(A_{IS}) (RF) (V_o)}$$

Where:

- $V_o$  = is the total volume of original sample in liters.

### **7.1.3 Surrogate Standards**

Surrogate standards, which are deuterated or halogenated compounds, are added to all samples and blanks during sample preparation. The recoveries of the surrogate compounds are used to isolate any problems that could occur throughout the entire analytical process. Three surrogates are used for VOAs. The acceptance criteria for the recoveries of the surrogate compounds are presented in Table 3-4.

### **7.1.4 Blanks**

The analysis of blanks is most important in the purge and trap technique used for VOAs since the purging device and the trap can be contaminated by residues from samples containing significant VOA concentrations or by vapors in the laboratory. Blanks are prepared by filling a sample bottle with low-organic water, which has been prepared by passing deionized water through an activated carbon column. Blanks are analyzed daily, prior to sample analysis.

### **7.1.5 Matrix Spike Duplicates**

At a frequency of one sample in 20 samples of similar matrix, matrix spike analysis is performed in duplicate for volatile organics. Matrix spikes are prepared by adding a known amount of standard to actual samples. For the volatile organic spiked fractions, two separate purge and trap concentrations are performed and analyzed as routine samples. The acceptance criteria for evaluating precision and spike recoveries are presented in Table 3-5.

## 7.2 Alcohols

The quality control procedures routinely employed by Gas Chromatography (GC) analyses of water for alcohols are presented below:

### 7.2.1 Standardization

The GC is calibrated initially with three standards covering the working range of the instrument.

Each day that analyses are performed, the instrument is standardized against a standard solution containing the compounds of interest. A Calibration Factor (CF) is determined by the Internal Standard (IS) method each time the standardization is performed. Internal standards, which are usually deuterated compounds, are added to all standards as well as to all blanks and samples before injection into the GC system. Internal standard areas for each sample are monitored to detect changes in instrument conditions.

The standardization calibration factor is calculated as follows:

$$CF = \frac{A_S}{I_S}$$

Where:

- $A_S$  = the integrated area of the characteristic peak for the compound in the standard.
- $I_S$  = the amount (ng) of the internal standard.

The concentration ( $C_0$ ) of the compound in the sample is calculated using the following equations:

$$C_o \text{ (ug/l)} = \frac{(A_x)}{(V_t) (CF)}$$

Where:

$A_x$  = the integrated area of the compound being measured.  
 $V_t$  = the total volume (ml) injected.

#### 7.2.2 Blanks

An aliquot of deionized water is taken through each preparation procedure each day samples are analyzed as a check for glassware and reagent contamination.

#### 7.2.3 Matrix Spike Duplicates

At a frequency of one sample in 20 samples of similar matrix, matrix spike analysis is performed in duplicate for the alcohols. matrix spikes are prepared by adding a known amount of standard to actual samples.

### 7.3 Metals

The quality control procedures routinely employed in inorganic chemistry analyses are presented below:

#### 7.3.1 Standardization

Precision and accuracy are an integral part of quality control, but they are only effective when instruments, solutions, and procedures have been standardized. Solutions are standardized by preparing standards of known purity and concentrations and using these standards to evaluate other solutions. Standards are either traceable to the National Bureau of Standards, or they are

certified by the manufacturers to contain a known concentration of analyte.

Standardization of instruments and methods is accomplished by preparing a series of standardized solutions and analyzing the standards according to a written procedure. From the results of the standards analyses, standard curves are constructed and used to determine the concentration of the species in each sample.

Standard curves are particularly useful in quantitative analyses using spectrophotometry. many spectrophotometric methods adhere to Beer's Law, which states that the adsorptivity of a substance is constant with respect to changes in concentration. For those calorimetric methods that adhere to Beer's Law and produce repeatable, stable color complexes, complete standard curves are performed at a minimum of once every 6 months. For metals analyses by atomic adsorption spectrophotometry, complete standard curves are performed each day that analyses for a particular metal are performed.

A number of methods follow Beer's Law, but for may reasons the standards are not reproducible from day to day, chemist to chemist, or reagent to reagent. When these methods are used, a complete set of standards is run either each day or with each set of samples.

#### **7.3.2 Verification Standards**

In general, methods that do not require a complete daily standard curve require the analysis of at least one standard each day to verify instrument and method performance. The results of the daily standard analyses must be within the control limits, which are set at the mean of 26 to 51 values (depending on the current number of available values)  $\pm 3$  times the standard deviation. Appropriate corrective measures must be taken if the acceptance criteria are not met.

### **7.3.3 Preparation Blanks**

An aliquot of deionized water is taken through each preparation procedure each day samples are prepared as a check for glassware and reagent contamination.

### **7.3.4 Duplicates**

One in 20 samples analyzed for a specific parameter is run in duplicate each day. Differing aliquots are used in many instances to conserve sample and to test for matrix interferences.

### **7.3.5 Matrix Spikes**

One in 20 samples analyzed for a specific parameter is spiked with the analyte each day, for those parameters for which a stable standard is available. An aliquot of standard solution is added to the sample.

## **7.4 Cyanide, pH, and Other Analytes**

The quality control procedures are detailed in Appendix E of the Laboratory's General Quality Assurance Plan.

## **8.0 DATA REDUCTION, VALIDATION, AND REPORTING**

Data reduction, validation, and reporting will be conducted as described below.

### **8.1 Data Reduction**

The calculation of final results from raw data varies from parameter to parameter with the calibration approach. The ratio of instrument response to analyte concentration is determined for one or more standards. In general, if the concentration/instrument response ratio is linear, the average of the ratios is used to calculate sample results. If the response is not linear, response is plotted against concentration, and sample results are quantitated from the resultant curve.

Results are generally expressed to two significant figures. Organic results for liquid samples are generally expressed in ug/L, while organic results for solid samples are expressed in ug/kg. Inorganic results for liquid samples are generally reported in ug/L or mg/L, while inorganic results for solid samples are expressed in mg/kg.

### **8.2 Data Validation and Reporting**

The results of quality control checks are the primary tools used for data validation. Quality control checks are described in Section 7.0. Acceptance criteria (control limits) are discussed in Section 3.0. Raw data and final results are reviewed by the laboratory group leader on a daily basis. The group leader confirms that documentation is complete and legible; qualitative identifications are accurate; calculations are accurate; results are expressed in the appropriate units and number of significant figures; and the required quality control checks were run and met

acceptance criteria. Review and approval of the data is documented by the group leader.

The "CLP-equivalent" deliverable and data validation will be performed on the DQO Level IV analyses only. DQO Level IV is considered legally defensible data and is sufficient to document compliance with LDR requirements. The analyses within DQO Level III are engineering study data and will be utilized solely within the treatability study and will not be validated.

The tabulated chemical-analytical data generated by the laboratory will be sent to the sampling coordinator who will log it into the validation tracking system. The data will be validated by the HALLIBURTON NUS Chemistry and Toxicology Department. Validation of the chemical-analytical data will include a quality assurance assessment to determine whether specified protocols were followed by the laboratory personnel. Results for field blanks and duplicates will be reviewed for consistency (i.e., relative percent difference values) and to identify laboratory artifacts. The laboratory will provide reagent blank, surrogate spike, and matrix spike results. This information will also be reviewed through comparison with the specified control limits (see Section 3.0). All validation will be performed using EPA's "Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses 2/88" and "Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analysis, 7/88" as a reference. The functional guidelines will be used in conjunction with additional criteria set forth by EPA Region VIII. Documentation of the validity of laboratory results will be provided to the HALLIBURTON NUS Project Manager in the form of letter reports.

Chemical-analytical data generated during the study will be reduced to a concise form for presentation in the Final report. The analytical results will be managed using an existing computer program developed by HALLIBURTON NUS specifically for chemical data



bases. This program is capable of handling a large number of chemicals and will be customized to accommodate all indicator parameters. Quality assurance procedures will be implemented to assure that no errors occur during data entry. The data entered into the program are checked by the computer operator, and the printouts are checked against the original laboratory sheets by a chemist.

## 9.0 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

Performance and systems audits are described in Sections QA-11 and QA-12 of the HALLIBURTON NUS Laboratory's General Quality Assurance Plan. The HALLIBURTON NUS Laboratory Services Group (LSG) participates in the following external performance audits in addition to their own internal blind quality control sample program. A copy of the results of these studies is available upon request.

- EPA Water Supply Studies (annually) -- Pittsburgh and Houston
- EPA Water Pollution Studies (annually) -- Pittsburgh and Houston
- EPA DMR-QA Studies (annually) -- Pittsburgh and Houston
- New York State DOH Non-Potable Water and Hazardous Waste Studies (semi-annually) -- Pittsburgh
- NIOSH PAT Rounds (quarterly) -- Houston
- EMSL-LV Radiochemistry Cross Check Studies (monthly) -- Pittsburgh
- Oklahoma Water Resources Board Studies (semi-annually) -- Houston
- Water and Wastewater Analysts Association (monthly) -- Houston

Certifications held by LSG are listed below:

- American Industrial Hygiene Association -- Asbestos by PLM, fiber count, metals, and organics accreditation (Houston)
- California Department of Health Services -- Hazardous waste certification (Pittsburgh)
- Florida Department of Health and Rehabilitative Services -- Wastewater certification (Houston)
- Kansas Department of Health and Environment -- Wastewater certification (Pittsburgh)
- National Voluntary Laboratory Accreditation program (NVLAP) -- Asbestos by PLM accreditation (Houston)
- New York Department of Health -- Wastewater and solid waste certification (Pittsburgh)
- North Carolina Department of Natural Resources and Community Development -- Wastewater certification (Pittsburgh)
- Nuclear Regulatory Commission -- Class B Broad Scope Materials License (Pittsburgh)
- Oklahoma Water Resources Board -- Wastewater certification program (Houston)
- Pennsylvania Department of Environmental Resources -- Drinking water certification (Pittsburgh)

- New Jersey Department of Environmental Protection -- Drinking water and wastewater certification (Pittsburgh)
- Texas Water Commission -- Wastewater analysis approval (Houston)
- Utah Health Laboratory -- Drinking water and wastewater certification (Houston)
- Water and Wastewater Analysts Association (Houston)

In addition, EG&G has audited the Pittsburgh laboratory for this project.

## **10.0 LABORATORY PREVENTIVE MAINTENANCE**

Preventive maintenance activities are described in Section QA-13 and Appendix J of the HALLIBURTON NUS Laboratory's General Quality Assurance Plan.

## **11.0 DATA ASSESSMENT FOR PRECISION, ACCURACY, AND COMPLETENESS**

Procedures to assess data quality are described in Section QA-8 of the HALLIBURTON NUS Laboratory's General Quality Assurance Plan.

## **12.0 SAMPLE, TREATED WASTE SPECIMEN, AND ANALYTICAL WASTE DISPOSITION**

The HALLIBURTON NUS Laboratory in Pittsburgh, Pennsylvania will be returning all treatability study samples, treated waste specimens, and wastes associated with the analysis of these samples and specimens to the Rocky Flats Plant. Wastewaters generated in the treatability study will be filtered and disposed of by the HALLIBURTON NUS Laboratory as discribed in the letter (C-28-91-PVF-1052) provided in Appendix I.

The Laboratory will handle the waste as discribed in AP-017. "Packaging and Shipment of Solar Pond Project Samples for Return to Rocky Flats" provided in Appendix I. Additional criteria that compliments this document is included below:

### **12.1 Segregation of the Wastes**

The laboratory will segregate the wastes as follows:

- Halogenated solvent waste
- Non-halogenated solvent waste
- Acid waste
- DI water waste
- Unused sample
- Treated waste specimens

### **12.2 Shipping the Wastes**

Prior to each return shipment, the following information will be faxed to J. D. Roberts:

1. Description of what is being shipped (number, type and contents of containers, etc.)
2. Name of the truck line transporting the material.

3. Date and material leaves your laboratory.

4. The estimated time of arrival at the Rocky Flats Plant.

The fax number of the Traffic Department is (303) 966-4588.

The shipping address of the plant is:

EG&G Rocky Flats, Inc.

Rocky Flats Plant

Rocky Flats, CO 80403

Attn: J. D. Roberts

Building 788



## REFERENCES

HALLIBURTON NUS, Deliverable 316, Onsite Laboratory Operating Instructions. Section RF-10. Revision 1.

HALLIBURTON NUS, 1991. Laboratory's General Quality Assurance Plan. Revision 1, 1989.

U.S. Department of Energy Nevada Operations Office and Reynolds Electric and Engineering Co., Inc. October 1988. Nevada Test Site Defense Waste Acceptance Criteria, Certification, and Transfer Requirements (NVO-325).

U.S. Environmental Protection Agency, February 1988. Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses.

U.S. Environmental Protection Agency, July 1988. Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses.

U.S. Environmental Protection Agency. Land Disposal Restrictions. 40 Federal Registry Part 268.

Water and Wastewater Technology, Mark J. Hammer. "Jar Tests," pages 36-29.

Weston, Roy F., July 1991. Sampling and Analysis of Solar Pond 207C Water and Sludge. Volume V.

**APPENDIX A**

**JAR TESTS**

## JAR TESTS

The effectiveness of chemical coagulation of water or wastewater can be experimentally evaluated in the laboratory by using a stirring device. The stirrer consists of six paddles capable of variable-speed operation between 0 and 100 rpm. In making tests, 1 liter or more of water is placed in each of the jars or beakers, and is dosed with different amounts of coagulant. After rapid mixing to disperse the chemicals, the samples are stirred slowly to promote floc formation and then are allowed to settle under quiescent conditions. The jars are mixed at a speed of 60 to 80 rpm for 1 min after adding the coagulant solution and then are stirred at a speed of 30 rpm for 15 minutes. After stopping the stirrer, the nature and settling characteristics of the floc are observed and are recorded in qualitative terms, as poor, fair, good, or excellent. A hazy sample indicates poor coagulation, while properly coagulated water contains floc that are well formed with the clear liquid between particles. The lowest dosage that provides good turbidity removal during a jar test is considered as the first trial dosage in plant operation. Ordinarily a full-scale treatment plant gives better results than a jar test at the same dosage.

For research or special studies, the beakers used in jar testing may be modified to more closely replicate actual mixing units constructed in treatment plants. Actual treatment plant operations may dictate a change in mixing and settling times to match those being used in water processing. Since other factors such as temperature, alkalinity, and pH influence coagulation, jar tests can also be run to evaluate these parameters and to determine optimum dosages under differing conditions. In addition to the affect of variation in water quality on chemical dosage, special studies can be conducted to measure optimum application of

coagulant aids, such as polymers or activated silica with the primary coagulant.

**APPENDIX B**  
**BULK SETTLING RATE TEST**

## **BULK SETTLING RATE TEST**

### **1. Apparatus**

- a. Either a 1 or 2 liter graduated cylinder.
- b. Timer

### **2. Procedure**

- a. Place feed waste, at the correct dilution and temperature, in a graduated cylinder. The correct dilution for a bulk settling rate determination is that dilution at which the demarcation between settling flocs and cloudy liquid can just be discerned. If a definite line forms as in zone settling, the sludge is too thick; if the only noticeable zone of solid-liquid demarcation is the buildup of settled floc on the bottom of the cylinder, the sludge is too thin. The coagulant/polymer concentration will be determined by the jar test. The room temperature should be investigated to investigate the expected extremes since temperature has a pronounced effect on settling rates. Also, this test should be run at several dilutions to make certain that the settling rate is determined at a point where solids concentration is not adversely affecting the bulk settling rate.
- b. Mix the sludge by moving a glass stirring rod with a rubber stopper at one end, up and down in the cylinder at a rate just sufficient to achieve complete sample homogeneity and then start the time.
- c. Record the time versus sludge volumes as the bulk of the

solids settle from 800 to 900 cc's to 400 to 500 cc's using any convenient sludge volume interval. If the sludge volume cannot be determined near the top of the cylinder, as is often the case, start the timer immediately after mixing the sludge and try to read the time at a sludge volume of 300 to 400 cc's.

- d. Plot the data as cylinder volume, ml vs. settling time, min. With the data points within upper and lower limits of the curve which form a straight line calculate the bulk settling rate (BSR) as follows:

$$\text{BSR(ft/hr)} = \frac{\Delta \text{ cc} \times 60 \text{ min/hr}}{\Delta \text{ min} \times (\text{cylinder calibration, cc/ft})}$$

where:

- 1) cylinder calibration for a one liter cylinder  $\approx$  883 cc/ft
- 2) cylinder calibration for a two liter cylinder  $\approx$  1482 cc/ft

$$\text{Plant Design Overflow Rate (ft/hr)} = 0.5 \times \text{BSR}$$

The overflow rate values calculated for data collected under conditions of maximum acceptable sample dilution are used for design purposes.

**APPENDIX C**  
**TEST PROCEDURE FOR PONDCRETE/SALTCRETE**  
**DISAGGREGATION TEST**



## **Test Procedure for Pondcrete/Saltcrete Disaggregation Test**

The disaggregation test requires a 1 kg representative sample of each as-is Pondcrete sample to be tested. The sample should not have been previously reduced in size, and if necessary can be a composite of similar materials. All of the weights requested and observations noted should be recorded on the attached data sheet.

1. If the material contains more than 5 to 10% moisture, remove the excess by filtration using a Buchner funnel and a medium pore, quantitative type filter paper. Measure the weight, volume, and TDS of the filtrate and hold the filtrate for possible metal analyses.
2. Place the 1 kg of sample (less than 10% moisture) in a preweighed drying pan, obtain a wet weight, and dry 12 to 14 hours in an oven maintained at 45° Celsius. Measure the dry weight of the sample.
3. Blend the dried sample by repeated passes through a Jones-type riffle splitter (minimum 5 passes) and then split the sample into two samples of approximately 500 grams each. Weigh each split.
4. Dry screen split "A" at the following U.S. Sieve (or Tyler equivalent) sizes: 75 mm (3"), 37.5 mm (1.5"), 19 mm (0.75"), 9.5 mm (0.375"), 4.75 mm (#4), 2 mm (#10), 0.85 mm (#20), 0.30 mm (#50), 0.15 mm (#100), 0.075 mm (#200). Weigh each size fraction including the -0.075 mm fraction. Report the weights of each size fraction along with the size distribution.
5. Place split "B" in a 2000 ml beaker and add twice the sample weight (approximately 1000 ml) of distilled water. Cover the

beaker with a plastic covering and secure with a rubber band. Allow the sample to set submerged for 24 hours, periodically observing the condition of the solids and solution (i.e., cracks developing in particles, particles disintegrating, color of solution, gas evolution, etc.).

6. Filter the solids as in Step 1.
7. Dry the solids as in Step 2.
8. Screen the solids as in Step 4.
9. Review the data sheet and make sure all of the data requested has been obtained, and all observations have been noted.

The above procedure is preferred, however, if obtaining a sufficient sample quantity is a problem, then the following procedure using only 500 grams of representative sample of each Pondcrete or Saltcrete sample can be used. All of the weights requested and observations noted should be recorded on the attached data sheet.

- A1. If the material contains more than 5 to 10% moisture, remove the excess by filtration using a Buchner funnel and a medium pore, quantitative type filter paper. Measure the weight, volume, and TDS of the filtrate and hold the filtrate for possible metal analyses.
- A2. Place the 500 grams of sample (<5 to 10% moisture) in a preweighed drying pan, obtain a wet weight, and dry 12 to 14 hours in an oven maintained at 45° Celsius. Measure the dry weight of the sample.

- A3. Dry screen split at the following U.S. Sieve (or Tyler equivalent) sizes: 75 mm (3"), 37.5 mm (1.5"), 19 mm (0.75"), 9.5 mm (0.375"), 4.75 mm (#4), 2 mm (#10), 0.85 mm (#20), 0.30 mm (#50), 0.15 mm (#100), 0.075 mm (#200). Weigh each size fraction including the -0.075 mm fraction. Report the weights of each size fraction along with the size distribution.
- A4. After weighing each size fraction, recombine them in a 2000 ml beaker and add twice the sample weight (approximately 1000 ml) of distilled water. Cover the beaker with a plastic covering and secure with a rubber band. Allow the sample to set submerged for 24 hours, periodically observing the condition of the solids and solution (i.e., cracks developing in particles, particles disintegrating, color of solution, gas evolution, etc.).
- A5. Filter the solids as in Step A1.
- A6. Dry the solids as in step A2.
- A7. Screen the solids as in Step A3.
- A8. Review the data sheet and make sure all of the data requested has been obtained, and all observations have been noted.

**PONDCRETE/SALTCRETE DISAGGREGATION TEST  
DATA SHEET**

**SAMPLE NUMBER:** \_\_\_\_\_  
**TEST START DATE:** \_\_\_\_\_  
**RESPONSIBLE TECHNICIAN SIGNATURE:** \_\_\_\_\_

1. WAS THE SAMPLE INITIALLY FILTERED? \_\_\_\_\_  
 IF "YES" -

FILTRATE WEIGHT \_\_\_\_\_ grams  
 FILTRATE VOLUME \_\_\_\_\_ ml  
 FILTRATE TDS \_\_\_\_\_ ppm  
 FILTRATE METAL ANALYSIS (YES/NO) \_\_\_\_\_  
 IF "YES" ATTACH ANALYTICAL REPORT

2. SAMPLE WEIGHT

PAN TARE WEIGHT \_\_\_\_\_ grams  
 PAN + WET SAMPLE \_\_\_\_\_ grams  
 PAN + DRY SAMPLE \_\_\_\_\_ grams  
 WET SAMPLE WEIGHT \_\_\_\_\_ grams  
 DRY SAMPLE WEIGHT \_\_\_\_\_ grams

3. WEIGHT OF EACH SPLIT

SPLIT A: \_\_\_\_\_ grams  
 SPLIT B: \_\_\_\_\_ grams

4. SCREEN ANALYSES

SIZE FRACTION	SPLIT A		SPLIT B (After Disagg.)	
	grams	wt%	grams	wt%
+75 mm				
-75 +37.5 mm				
-37.5 +19 mm				
-19 +9.5 mm				
-9.5 +4.75 mm				
-4.75 +2 mm				
-2.0 +0.85 mm				
-0.85 +0.30 mm				
-0.30 +0.15 mm				
-0.15 +0.075 mm				
-0.075 mm				
TOTAL				

DISAGGREGATION TEST DATA SHEET  
PAGE 3

SAMPLE NUMBER: \_\_\_\_\_

5. SPLIT B WEIGHT (from 3, above): \_\_\_\_\_ grams  
VOLUME OF DISTILLED WATER ADDED: \_\_\_\_\_ ml  
START DATE AND TIME: \_\_\_\_\_

OBSERVATIONS: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

FINISH DATE AND TIME: \_\_\_\_\_

6. FILTER SPLIT B AFTER IMMERSION IN DISTILLED WATER

FILTRATE WEIGHT \_\_\_\_\_ grams  
FILTRATE VOLUME \_\_\_\_\_ ml  
FILTRATE TDS \_\_\_\_\_ ppm  
FILTRATE METAL ANALYSIS (YES/NO) \_\_\_\_\_  
IF "YES" ATTACH ANALYTICAL REPORT

7. SPLIT B WEIGHT AFTER FILTRATION

PAN TARE WEIGHT \_\_\_\_\_ grams  
PAN + WET SAMPLE \_\_\_\_\_ grams  
PAN + DRY SAMPLE \_\_\_\_\_ grams  
WET SAMPLE WEIGHT \_\_\_\_\_ grams  
DRY SAMPLE WEIGHT \_\_\_\_\_ grams

8. SCREEN ANALYSIS - REPORT ABOVE IN TABLE

**APPENDIX D**  
**PROCEDURE FOR THE LAROX LABORATORY FILTER**  
**LABOX 25**

## PROCEDURE FOR THE LAROX LABORATORY FILTER LABOX 25

### CHOICE OF FILTER CLOTH:

The Labox filter comes with two different cloth fabrics. When an extremely pure filtrate is desired, the best choice for filter cloth is No. 71-2209K3. For high capacity and low moisture, No. 71-2155 is recommended.

### HOW TO USE THE FILTER:

Place the filter cloth of your choice over grid in filtrate vat. Place sealing on top of the cloth, fasten cylinder into filtrate vat by turning it and tighten the joint manually.

### PUMPING:

Pour mixed slurry into cylinder (maximum 150 ml of slurry).

Put cylinder and piston between moving pressing plate and upper pressing plate so that the claws go into corresponding grooves located in the upper and lower part of the cylinder-piston unit. Make sure that air on/off valve is closed.

Set the pumping pressure in pressure regulator (1.5 bar = 4 bar pumping pressure). Place filtrate vat underneath filtrate hose. To start pumping turn cylinder drive valve latch up. Continue pumping cycle as long as cylinder moves.

If the thickness of the cake thus formed seems satisfactory, proceed to PRESSING cycle. However, if the cake thickness is not suitable, press drive valve cylinder latch down, whereupon cylinder goes down. Now you can remove the cylinder and piston, and add some

slurry in the cylinder. To get a desired cake thickness, repeat pumping cycle as many times as necessary.

#### PRESSING:

Set pressure in pressure regulator to match the desired compressing pressure (e.g. 6 bar= 16 bar compressing pressure). Turn cylinder drive valve latch up, and set stopwatch to measure the time used for pressing time. To end pressing cycle turn cylinder drive valve latch to OFF. If cake wash is desired, go to CAKE WASH.

#### AIR DRYING:

Push in limit knob for cylinder movement and start air drying. Turn air distribution valve to AIR BLOW and open air on/off valve by turning it carefully about 1/4 of a turn. The cylinder starts now to go down, stopping against limiter. When air starts to come out of filtrate hose, set watch to take the time used for drying. To stop air drying turn first air distribution valve to AIR OUT, whereupon pressure is released from the chamber. After this turn air on/off valve off.

#### CAKE DISCHARGE:

To remove the cake turn first cylinder drive valve latch up for a moment. After cylinder has moved up a little, turn drive valve latch to OFF and pull limit knob out. then turn drive valve latch down; now the cylinder will go all the way down. Take cylinder out of the filter, remove filtrate vat from cylinder and using cake discharge piston and cake discharge cup, take the cake out of the cylinder.

If the cake is too stubborn to be removed manually, use pressure



air. Put the cylinder, cake discharge piston and cake discharge cup between the moving pressure plate and upper pressure plate. Then turn cylinder drive latch up. The piston will now push the cake into the cake cup.

#### CAKE WASH:

Cake wash is normally performed after a short I (first) Pressing cycle. Turn cylinder drive valve to down position. After the cylinder is down, take it in your hand, and pour a measured amount of wash liquid over the cake. Put the cylinder and piston back in place. Run a cake washing cycle the same way as the pressing cycle. When most of the wash liquid has penetrated through the cake, proceed to PRESSING. the cake wash cycle as well as the pumping cycle may be repeated several times.

#### PRECAUTIONS:

- DO NOT RUSH
- DO NOT STICK YOUR FINGERS IN BETWEEN MOVING PARTS
- ONLY HANDLE ONE VALVE AT A TIME
- PROTECT YOURSELF PROPERLY, E.G. WEAR A MASK WHENEVER YOU ARE HANDLING HAZARDOUS MATERIALS
- MAXIMUM PRESSURE OF COMPRESSED AIR IS 7 BAR

#### CALCULATIONS AND DIMENSIONING:

After a test run weigh the cake and measure its moisture content. Measure also the filtrate amount.

Total time T (minutes) = time used for pumping + pressing I + cake wash + pressing II + air blow drying + 4 minutes.

Because pumping is in the filter takes place in stages, the time used for pumping must be estimated. With easy-to-filter materials pumping time is estimated at one minute, and with hard-to-filter substances at from 2... 5 min.

Capacity C (kg/m<sup>2</sup>h)

$$C = \frac{(W \quad W \times M/100) \times 60}{0.0025 \times T} \quad (\text{kg/m}^2)$$

W = weight of cake (kg)

M = cake moisture (%)

T = total cycle (min)

Slurry capacity Cs (l/m<sup>2</sup>h)

$$Cs = \frac{Q \quad \times \quad 60}{T \times 0.0025} \quad (\text{l/m}^2\text{h})$$

Q = amount of slurry (l)

T = total cycle (min)

#### MAINTENANCE:

Wash the parts that have come in contact with the slurry after each test run. If necessary, to clean the air channel the air control valve can be unscrewed from the piston. At the same time you can check the condition of the O-ring (dia 8 x 2.5). After the wash, check the lip sealing of the piston (G1-45 x 57 x 6 nitrile).

**APPENDIX E**

**TEST PROCEDURE FOR FANN V-G VISCOMETER**

**(OPERATING INSTRUCTIONS AND PRODUCT INFORMATION)**

## OPERATING INSTRUCTIONS FOR MODEL 35 FANN V-G METER

### GENERAL

1. Connect instrument to 115 volt, 60 cycle, A. C. power source.
2. Speeds, rotor sleeve: **CHANGE GEARS ONLY WHEN MOTOR IS RUNNING.**  
600 rpm - Gear shift knob down, motor switch at "high"  
300 rpm - Gear shift knob down, motor switch at "low"  
200 rpm - Gear shift knob up, motor switch at "high"  
100 rpm - Gear shift knob up, motor switch at "low"  
6 rpm - Gear shift knob center, motor switch at "high"  
3 rpm - Gear shift knob center, motor switch at "low"

### PLASTIC VISCOSITY AND YIELD VALUE

1. Place recently agitated sample in test cup furnished. A line scribed inside the cup at 350 cc level is for barrel equivalent volume. With rotor sleeve running at 600 rpm, wait for dial to come to steady value. Note dial reading.
2. Set motor switch at low and obtain 300 rpm reading in the manner outlined in step one.
3. 600 reading - 300 reading equals plastic viscosity (p. v.) in cps.  
300 reading - (p. v.) equals yield value (y. v.) in lbs./100 sq. ft.

### GEL STRENGTH

1. Stir sample thoroughly at 600 rpm.
2. Change gear shift to center then turn motor off.
3. Allow desired rest time (10 seconds to 10 minutes) then turn switch to low (3 rpm).
4. Read dial at instant of gel break. Reading is in lbs./100 sq. ft.

### CONSISTENCY CURVES AND THIXOTROPIC STUDIES

1. For a more complete consistency curve take readings at 200, 100, and 6 rpm in addition to the above. Plot results, speed vs. dial deflection, and extrapolate straight portion of curve to stress axis to obtain yield value intercept.
2. Thixotropic studies are usually made by taking a series of readings beginning at slow speed on unstirred sample. Measurements are made at successively higher speeds to maximum then the downcurve is run immediately. Plotted, the hysteresis loop formed by the upcurve and downcurve is a measure of thixotropy.

Clean instrument by running at high speed with rotor sleeve immersed in water or other solvent. Remove rotor sleeve by twisting slightly to release lock pin. Wipe bob and other parts thoroughly.

# INTERCHANGABLE TORSION SPRINGS FOR MODELS 34, 35 and 35S, FANN V-G METERS

## MAXIMUM MEASURABLE VISCOSITIES - Cps (With standard bob and rotor combination)

		Standard							
Spring No.	Spring Constant Kg	27S	25S	25D	22S	21S	22D	20S	20D*
Range Factor		77.4	193.5	387	774	1161	1548	1935	3870
		1/5	1/2	1	2	3	4	5	10
Rotor RPM	600	30	75	150	300	450	600	750	1500
	300	60	150	300	600	900	1200	1500	3000
	200	90	225	450	900	1350	1800	2250	4500
	100	180	450	900	1800	2700	3600	4500	9000
	6	3000	7500	15,000	30,000	45,000	60,000	75,000	150,000
	3	6000	15,000	30,000	60,000	90,000	120,000	150,000	300,000

\*For Use Only with special closed-end rotor

TO USE: Install desired spring in instrument and operate in normal manner. Multiply dial reading by range factor.

NOTE: All springs are calibrated at the factory but for best accuracy, calibration should be rechecked after installation, by dead weight method or by known fluids.

Formula for calculating Kg:

$$K_s = \frac{G g R}{\Theta} \quad \text{where:} \quad \begin{array}{l} K_s = \text{Spring Constant - Dyne cm/deg.} \\ G = \text{Load in grams} \\ g = 981 \\ R = \text{Radius arm - cm} \\ \Theta = \text{Dial reading} \end{array}$$

Wrap light string or thread around bob or other cylinder of known diameter fastened to bob shaft.  
Run string over a pulley and hang weights on string to deflect spring. Adjust free length of spring to get desired deflection.

FANN INSTRUMENT CORPORATION

P. O. Box 6101

Houston, Texas

**fann****OPERATING INSTRUCTIONS - FANN VISCOMETERS**

*V. G. Muter  
Rec'd Oct 3, 1973  
#1 Spring*

**GENERAL**

1. Connect instrument to proper power source.
2. On the 35 models refer to nameplate for speed changing instructions.
3. The two-speed models have a gear shift knob at top center of gear case. Pull knob up for 300 rpm — push down for 600. Center position is neutral. A switch at left side of gear case on Model 34A provides a stirring speed.
4. When cranking the HC34A, turn fast enough so clutch can be felt slipping at all times.
5. All models have a hand knob for gel measurements. This knob should be at neutral for determining gels.
6. Change gears while motor is running to obtain desired speeds. Read torque or shear stress values from dial in instrument head.

Model No.	Volts	Hz.	Amps	Rotor Speeds — rpm
35A	115	60	.75	3, 6, 100, 200, 300 & 600
35SA	115	50	.60	3, 6, 100, 200, 300 & 600
34A	12	AC-DC	3.5	300, 600 and stirring speed
HC34A		Handcranked		300 and 600

**Notes:** 1. The SR-12 Gear Box Attachment, which can be fitted to the 35 series instruments, provides the following additional speeds.

0.9, 1.8, 30, 60, 90 & 180 rpm

2. Transformers are available for 230:115 volts and 115-230:12 volts.

7. Twelve rotor-bob combinations plus eight different torsion springs are available. These fit all models giving a total of ninety-six different measuring systems as described on pages 3 and 4.
8. Oiling or greasing of viscometer is not required in normal service. The bob and rotor should be cleaned after each test and examined periodically for dents, abrasion or other damage. Accuracy is not possible unless these components are in good mechanical condition and turn without wobble.
9. Rotors can be removed from socket by twisting CCW while gently pulling straight down. Bob shaft end is tapered and fits a matching hole in bob. Twist, while pulling downward, to remove. Bobs B1 and B2 are made hollow to lessen weight and should not be heated above 200°F without adequate precautions.

**OPERATION**

1. Sample cups have a line at the proper level to hold 350 ml. Fill to this point with recently stirred test fluid. A scribed line on rotor indicates proper immersion depth which should not be exceeded. If other sample holders are used, the space between bottom of bob and bottom of cup should not be less than 0.5" (1.27 cm).
2. Turn motor on, select desired speed and read shear stress values from dial.

**fann****fann instrument corporation**

P. O. Box 36423 • 8625 Meadowcroft Street • Houston, Texas 77042  
Cable address: FANN Telephone (713) 781-5663

## FLOW MODELS

Figure 1 shows typical flow curves for four important flow models. The multiple-speed models are useful in constructing such rheograms to classify unknown fluids.

### 1. Newtonian Flow Model

As shown by A, Figure 1, the relationship between shearing stress and shear rate is directly proportional. Only one point is required to define this curve.

Table 1 may be used to quickly determine Newtonian viscosities with any of the rotor-bob-spring combinations. Newtonian viscosity in cp may be read directly from the dial of any model viscometer run at 300 rpm with the R1-B1-F1 combination, or other springs may be used providing dial reading is multiplied by "f" factor.

### 2. Bingham Plastic Flow Model

Run the viscometer at 600 rpm, using the R1-B1-F1 components, until dial reaches a steady value. Note dial deflection as  $\phi$  600. Change to 300 rpm and read  $\phi$  300. A spring other than F1 may be used if the dial readings are multiplied by proper "f" factor, but other combinations of R-B can not be used for this rapid, two-point method.

Then,

$$\phi 600 - \phi 300 = \eta_{pl} = \text{plastic viscosity in cp}$$

and,

$$\phi 300 - \eta_{pl} = \text{yield value in lb./100 ft.}^2$$

Gel strengths are measured after thorough stirring of sample at 600 rpm. On 35 series models, after stirring, set gears quickly for 3 rpm, then turn motor off. After desired rest period, switch motor on and read dial at instant of gel break. The 34 series models use a hand knob for this measurement. Turn knob slowly in direction of indicator and read as above. Gel readings are in lb./100 ft.<sup>2</sup>.

Other rotor-bob combinations may be used, provided shear rates are calculated for the material being studied. This is particularly true when combinations resulting in the larger gap sizes are used.

### 3. Pseudoplastic Flow Model

The apparent viscosities of these fluids decrease as shear rates increase. Most industrial fluids fall within this classification.

An empirical relation known as the power law is most often used to characterize fluids of this type. A log-log plot of shear stress vs. shear rate is constructed to obtain constants K and n. K is the intercept of the flow curve on the stress axis at unity rate of shear, and is a measure of consistency. The higher K, the more viscous the fluid. n, the slope of the line, is a measure of non-Newtonian behavior, and for pseudoplastics, falls between zero and unity.

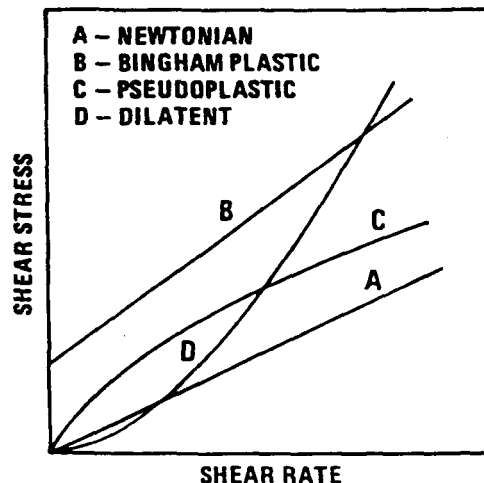
As with the previous flow model, use of rotor-bob combinations giving large gap sizes can lead to serious errors unless corrections are made.

### 4. Dilatent Flow Model

Methods for this model are often the same as for pseudoplastics, the difference being that apparent viscosities increase with increasing shear so n is always greater than 1.

Experimental data from these viscometers may be manipulated according to any of the accepted techniques for concentric cylinder types. Many books on the subject of rheology are available to the serious researcher.

Fig. 1



## TO CHANGE AND CALIBRATE TORSION SPRINGS

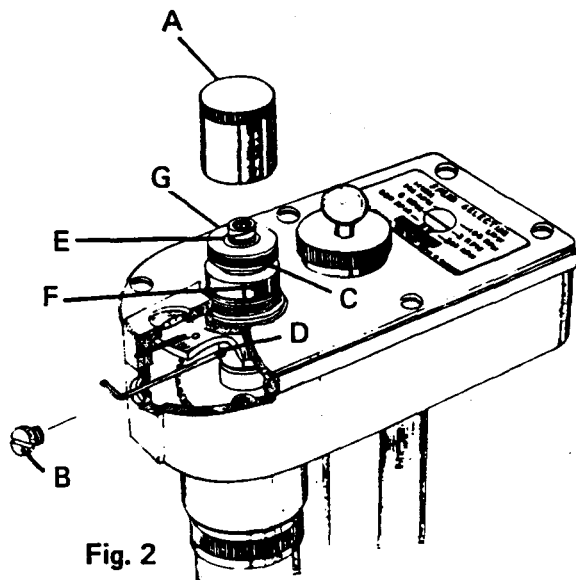


Fig. 2

1. Remove dust cap A and plug screw B.
2. Loosen set screws C and D about 1/2 turn. Spring can be lifted out. Do not stretch spring.
3. Insert new spring, making sure bottom mandrel is properly oriented and seated. Holding pressure of set screw D, acting through a bottom clamp ring similar to E should bear against the point at which spring leaves threaded mandrel. A notch cut into upper end of bottom mandrel will help to locate this point. Top of upper threaded mandrel inside spring should be exactly flush with clamp ring E. Tighten set screws.
4. Set screw F can be loosened, permitting knob G to be turned so dial can be zeroed under index. G is also movable vertically, enabling the spring to be clamped in a "free" position, neither stretched nor compressed.
5. Remove rotor and bob; then clamp calibrating fixture DW 3 to instrument legs. Place the 2 cm diameter aluminum spool on bob shaft, slip knotted end of thread into slotted hole; wrap once around spool, then over pulley. Position fixture so thread is horizontal. Hang weights on thread as described below and adjust spring constant if necessary.

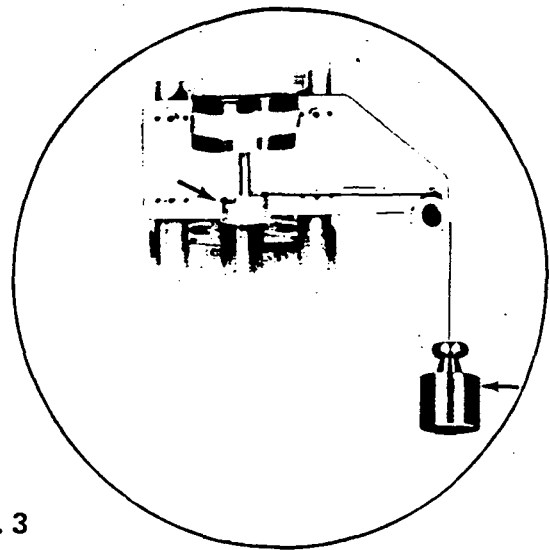


Fig. 3

DW3  
Calibration Fixture

Formula for Calculating Spring Constant  $K_s$

$$K_s = \frac{Ggr}{\phi}$$

where  $K_s$  = Spring constant — dynes/cm/deg.

G = Load in grams

g = 981

r = Radius arm = 1 cm

$\phi$  = Dial reading — degrees

### EXAMPLE:

From experience we know that the required setting for the F1 spring is 386 dynes/cm/degree with the R1-B1 combination.\* Using the 50 gm weight supplied with fixture, we have,  $\frac{50 \times 981 \times 1}{386} = 127^\circ$ .

Test material, undergoing shear in the annulus, imparts torque not only to bob wall but also, to some degree, depending on gap width, to ends of bob. In this case, the spring is made 6% stiffer to compensate for this additional load.

\*The effective  $K_s$  is less, 363 dynes/cm/degree. This is caused by "end effects" on bob and rotor.

### Rotor-Bob Dimensions

Unit	r — cm	Length — cm	Cyl. Area-cm <sup>2</sup> x Radius-cm
B1	1.7245	3.8	71.005
B2	1.2276	3.8	35.981
B3	.86225	3.8	17.751
B4	.86225	1.9	8.876
R1	1.8415		
R2	1.7589		
R3	2.5867		



## DIMENSIONS — ROTORS — BOBS — SPRINGS

### Rotor-Bob Shear Rates

Comb.	sec. $^{-1}$ /Rev.	$R_r/B_r$	Gap-cm
R1 — B1	1.7034	1.0678	.1170
R1 — B2	.37723	1.5001	.6139
R1 — B3	.26845	2.1357	.9793
R1 — B4	.26845	2.1357	.9793
R2 — B1	5.41066	1.0199	.0344
R2 — B2	.40865	1.4328	.5313
R2 — B3	.27589	2.0399	.8967
R2 — B4	.27589	2.0399	.8967
R3 — B1	.37723	1.5000	.8622
R3 — B2	.27052	2.1071	1.3591
R3 — B3	.23579	3.0000	1.7245
R3 — B4	.23579	3.0000	1.7245

### Torsion Springs

No.	Factor f	Dynes/cm/deg.*
F0.2	0.2	77.2.
F0.5	0.5	193
F1	1	386
F2	2	772
F3	3	1158
F4	4	1544
F5	5	1930
F10	10	3860

\*With R1-B1 Combination

**TABLE 1**  
**TO RAPIDLY DETERMINE NEWTONIAN VISCOSITIES**  
**IN cp. WITH FANN VISCOMETERS**

ROTOR rpm	SPEED FACTOR S
.9*	333.3
1.8*	166.6
3	100
6	50
30 *	10
60 *	5
90 *	3.33
100	3
180 *	1.667
200	1.5
300	1.0
600	.5

#### FORMULA:

$$\eta_N = S \times \phi \times f \times C$$

where,

S = Speed factor

$\phi$  = Dial reading

f = Spring factor

C = Rotor-bob factor

$\eta_N$  = Newtonian viscosity - cp.

EXAMPLE: Using an R2 — B1 combination at a speed of 30 rpm with an F5.0 spring, dial deflects to 189.  
 $10 \times 189 \times 5 \times .315 = 2977$  cp.

ROTOR-BOB COMB.	R-B FACTOR C
R1 — B1	1.000
R1 — B2	8.915
R1 — B3	25.392
R1 — B4	50.787
R2 — B1	.315
R2 — B2	8.229
R2 — B3	24.707
R2 — B4	49.412
R3 — B1	4.517
R3 — B2	12.431
R3 — B3	28.909
R3 — B4	57.815

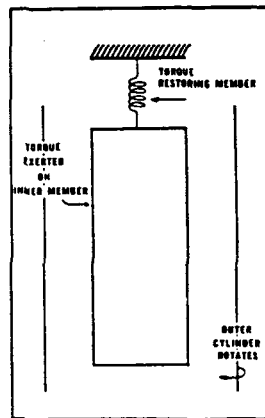
NOTE: C values are calculated from rotor-bob dimensions. Combinations with the larger gaps are likely to give results that differ from these figures. For best accuracy, calibration with a standard fluid having a viscosity near the range in which you are working is recommended for each combination.

\*Additional speeds available  
 with SR-12 Gear Box Attachment.  
 Fits Models 35, 35A, 35S and 35SA.

# FUNDAMENTAL RHEOLOGICAL CONCEPTS

Rheology is that branch of physics dealing with the deformation and flow of matter; consequently an understanding of this science is vital to a great number of manufacturing processes and industrial operations. Rheological properties are relied upon for control of the hydrodynamic properties of the mud and cement used in the drilling of an oil well. Rheological properties are important in converting a shapeless slip into a delicate sculptured object, and in extruding a plastic into a mold. The lubrication of an internal combustion engine, distribution of ink on a high speed printing press, and the control of the brushing, spraying, and leveling qualities of a household paint represent but a few of the many applications for measurement of rheological properties.

The line of FANN V-G METERS has been engineered to meet the need for accurate measurement of fundamental rheological properties. These viscometers are used in petroleum production and refining, and in the manufacture or processing of adhesives, ceramics, foods, paints, and numerous other products. They are designed to give years of dependable service regardless of the operational environment.



## BASIC OPERATING PRINCIPLE OF THE FANN V-G METERS

The Fann V-G Meters are rotational-type viscometers. Fluid is contained in the annular space between two coaxial cylinders. The outer cylinder, or rotor, suspended in precision bearings, is driven at a constant rotational velocity and the torque arising from the fluid's viscous drag is exerted on the inner cylinder, or bob. The torque is rapidly balanced by a helically wound spring and the deflection is read on a calibrated dial through an optical reticule.

In this viscometric method, flow data are best analyzed in terms of the fundamental relationship between shear rate, proportional to rotor rpm, and shearing stress, proportional to dial reading.

The Fann V-G Meters are properly designed rotational viscometers in that the cylindrical rotor and bob design permits calculation of the shear rate corresponding to each rotational velocity.

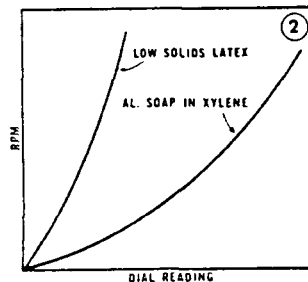
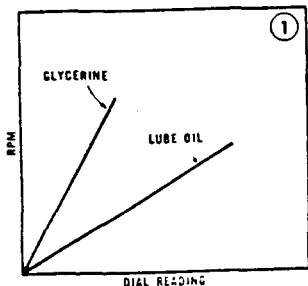
These calculations provide criteria for identifying flow type, maintaining adequate process control, and scaling up laboratory studies for a process prototype. In addition, the entire material can be homogeneously sheared and equilibrium flow conditions achieved rapidly at each shear level; uniform shear rate gradients are produced, and time-dependent properties can be readily analyzed.

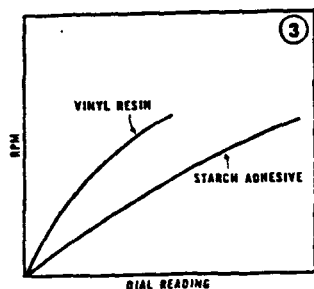
## SOME REPRESENTATIVE RHEOLOGICAL MODELS

Four types of ideal flow models are frequently used in rheology, viz: the Simple Newtonian, and the following Non-Newtonians: Pseudoplastic, Flow with Shear Rate Thickening, and Bingham Plastic. A very brief survey of the flow characteristics of each is given here although it is recognized that the properties of a material are frequently determined not by a

single ideal model but by a complex of rheological properties.

*The Simple Newtonian* is characterized by a straight line flow curve through the origin for all values of shearing stress (Fig. 1). This means that shear rate is directly proportional to shearing stress. The proportionality constant, called the coefficient of viscosity, is





calculated from the reciprocal slope of the flow curve. Examples are water, glycerol solutions, certain oils, etc.

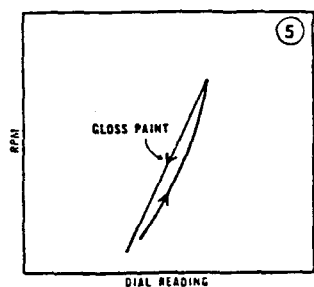
*Non-Newtonian* is applied to a material whose viscous resistance

is a function of the flow conditions. Examples are: adhesives, paints, printing inks, drilling fluids and cements, polymeric materials, and food products. (Viscous resistance which is also a function of time is discussed under the section on Thixotropy.)

A single point "viscosity" measurement, such as timing a given volume of material through an orifice or measuring the force required to rotate a paddle in the material at some arbitrary chosen rotational speed has little fundamental significance for the Non-Newtonian. The single point "viscosity" determination does not represent true flow behavior but is instead a single apparent viscosity out of an infinite spectrum of apparent viscosities. In order to characterize the flow behavior of these systems flow resistance must be measured at a minimum of two known shear rate levels.

A *Pseudoplastic* system flows at any finite stress with viscous resistance decreasing to some limiting value as the shear intensity increases (Fig. 2). Some investigators refer to this model as liquid flow with shear rate thinning.

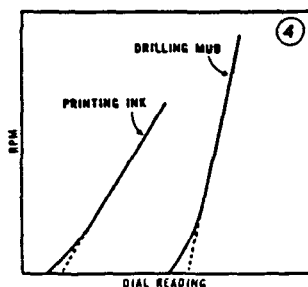
*Flow with Shear Rate Thickening* is the model name for systems exhibiting an increased viscosity with increasing shear intensity (Fig. 3). In some systems the effect may appear only above a minimum critical value. The term dilatancy is also used to describe this model.



Some investigators use power-type equations to empirically fit the flow curves of the above discussed Non-Newtonians and plot the log of shear rate versus the log of shearing stress. Very often a straight line is obtained

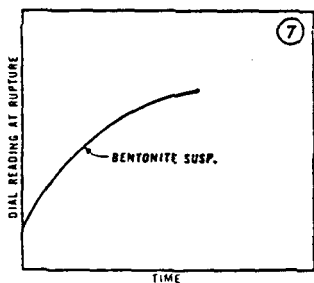
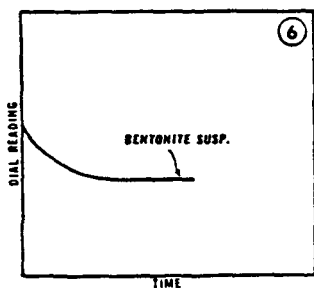
for a wide range in data with the result that the slope ( $d \log \text{shear rate} / d \log \text{shearing stress}$ ) can be related to a "structure number" or "index of flow type." For example the "structure numbers:  $>1$ ,  $<1$ ,  $=1$ , denote Pseudoplastic, Flow with Shear Rate Thickening, and Simple Newtonian, respectively. These "structure numbers" are used in a variety of hydrodynamic calculations.

The *Bingham Plastic* is illustrated in Fig. 4. Flow resistance in this system is characterized by two parameters, plastic viscosity, and yield point. The yield point intercept indicates that flow can start only if a shearing stress larger than the yield point is applied. Plastic viscosity is calculated from the reciprocal slope of the curve.

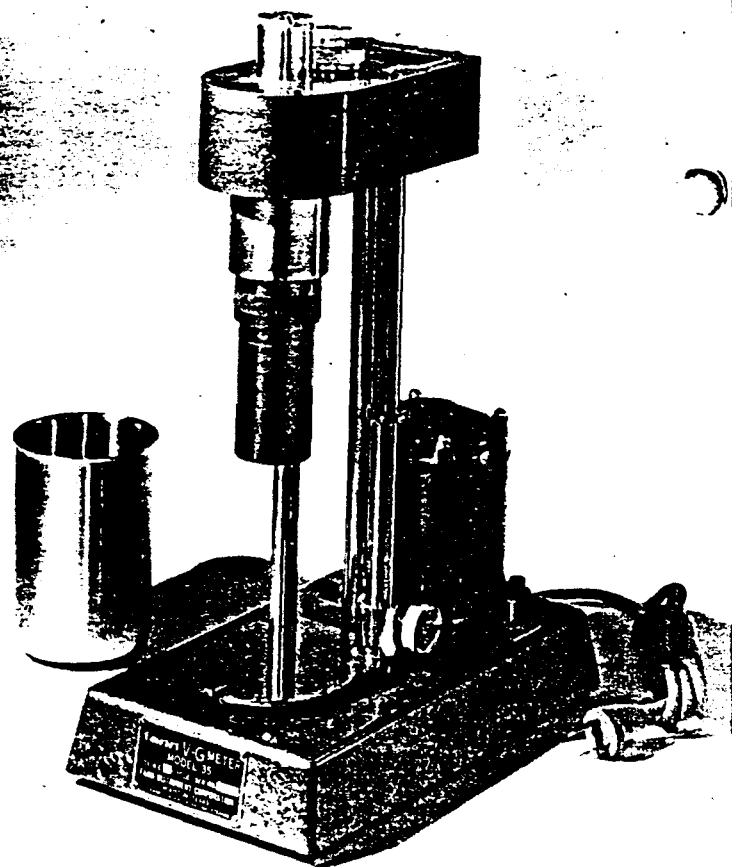


*Thixotropy*—Many processes involve the handling and production of materials which, in addition to exhibiting anomalous flow behavior as a function of flow conditions, are also affected by the parameter of time. There exists a class of materials

which are fluid under shear but become gel-like when left undisturbed. This softening under shear followed by a time-dependent return to the harder state is termed Thixotropy. It is evidenced under ideal conditions in a rotational viscometer by the appearance of a hysteresis loop in the shear rate-shearing stress curve (Fig. 5) and also by a gradual relaxation or recovery of stress, with time, at a constant shear level (Fig. 6). In using these methods care must be exercised to exclude temperature or degradation effects since they might be mistaken for the isothermal-reversible thixotropic process. The development of gel structure with time under static conditions is further evidence of the presence of the phenomenon (Fig. 7).



## STANDARD MODELS OF THE FANN V-G METER



# MODEL 35 FANN V-G METER

A research caliber, precision-built viscometer, the Model 35 is driven by a 115v—50 or 60 cycle dual speed synchronous motor. Rotor speed changes for 3, 6, 100, 200, 300, and 600 rpm are effected smoothly and rapidly without ever stopping rotation. The Model 35 is supplied with our standard chrome plated rotor-bob combination. Special rotor-bob combinations for supplementing shear rate and viscosity levels, or for use with corrosive liquids are readily available at extra cost. A wide selection of torsion springs which can be easily interchanged are stocked. Custom built constant temperature baths are also available.

Instrument finished in brown hammertone and polished chrome. A stainless steel sample cup, Part No. 3560, equipped with pins to locate and lock to movable platform, is included.

When referred to our standard rotor-bob

combination the following average shear rates and maximum measurable viscosities are obtainable at the several speed settings:

SPEED SETTING	AVERAGE SHEAR RATE, SEC. <sup>-1</sup>	MAXIMUM MEASURABLE VISCOSITY, CP.
600	959	150
300	480	300
200	320	450
100	160	900
6	9.6	15,000
3	4.8	30,000

This wide spectrum in shear rates available with a single standard rotor-bob combination is invaluable for the establishment of relationships between the rheological models discussed above. Data related to thixotropic processes may be readily obtained at the 3 and 6 rpm shear rate levels.

### ACCESSORIES

Part No.	
3583	Hardwood carrying case
3584X	230-115v transformer

### MOTOR CHARACTERISTICS

Model	Voltage	Frequency	Amperes	Motor h. p.
35	115	60 cycle	.5	1/75
35S	115	50 cycle	.5	1/100

### DIMENSIONAL DATA

Model	Height	Width	Depth	Weight
35 and 35S	15 1/2"	6"	10 1/2"	16 lbs.

### BIBLIOGRAPHY

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- Proceedings International Rheological Congress (Holland—1948)*, North-Holland Pub. Co., Amsterdam.
- Pryce-Jones, J., "Studies in Thixotropy," *Kolloid Feit.*, 129, 96 (1952).
- Reiner, M., *Deformation and Flow*, H. K. Lewis and Co., Ltd., (1949).
- Savins, J. G., and Roper, W. F., "A Direct-Indicating Viscometer for Drilling Fluids," *American Petroleum Institute, Drilling and Production Practice*, 7 (1954).

## FEATURES COMMON TO MODELS 34 AND 35 FANN V-G METERS

(With standard rotor-bob and spring combination)

Direct centipoise reading of viscosity at the 300 rpm setting. Simple multiplying factors for other speed settings.

Rapid determination of the Bingham Plastic parameters, Plastic Viscosity and Yield Point from the 600 rpm and 300 rpm settings:

PLASTIC VISCOSITY (cp.) = 600 rpm Reading — 300 rpm Reading

YIELD POINT (lb./100 ft.<sup>2</sup>) = 300 rpm Reading — Plastic Viscosity

Designed to meet the rough usage and portability requirements of "on-the-spot" field and process control work, this instrument retains most of the precision features of the Model 35. It is available as either a 6 or 12 volt AC DC model. A constant-speed governor controlled motor and gear train combination drives the rotor at 600 rpm and 300 rpm. Rotor speed changing between these settings is effected smoothly and rapidly without stopping rotation. The Model 34 is supplied with our standard chrome plated rotor-bob combination. Special torsion springs and rotor-bob combinations for supplementing shear rate and viscosity level, or for use with corrosive liquids, are readily available at extra cost. A choice of either the No. 3467 or 3468 connecting cord is furnished with instrument.

Other unique features of the Model 34 include:

Blinker light to safeguard against low supply voltage.

Knurled handwheel for the evaluation of thixotropic processes.

Governor release switch to permit high intensity shearing before measurements.

When referred to our standard rotor-bob combination the following average shear rates and maximum measurable viscosities are obtainable:

SPEED SETTING	AVERAGE SHEAR RATE, SEC. <sup>-1</sup>	MAXIMUM MEASURABLE VISCOSITY, CP.
600	959	150
300	480	300

## MODEL FANN V-G METER

### MOTOR CHARACTERISTICS

Model	Voltage	Amperes	Motor h. p.
34	6 AC-DC	6	1/50
34	12 AC-DC	3	1/50

### DIMENSIONAL DATA

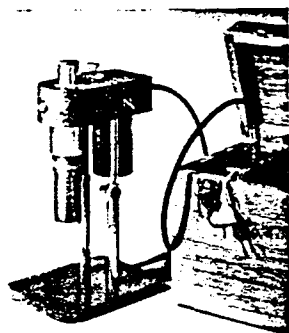
Model	Height	Width	Depth	Weight
34	9 1/2"	5 1/2"	7"	7 1/2 lbs.

(12" extended)

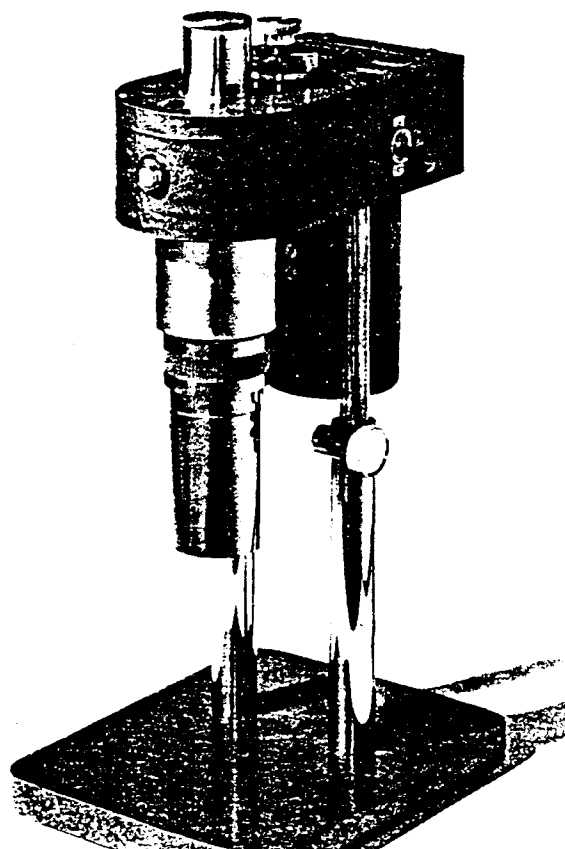
### ACCESSORIES FOR MODEL 34-6 and 12 volt

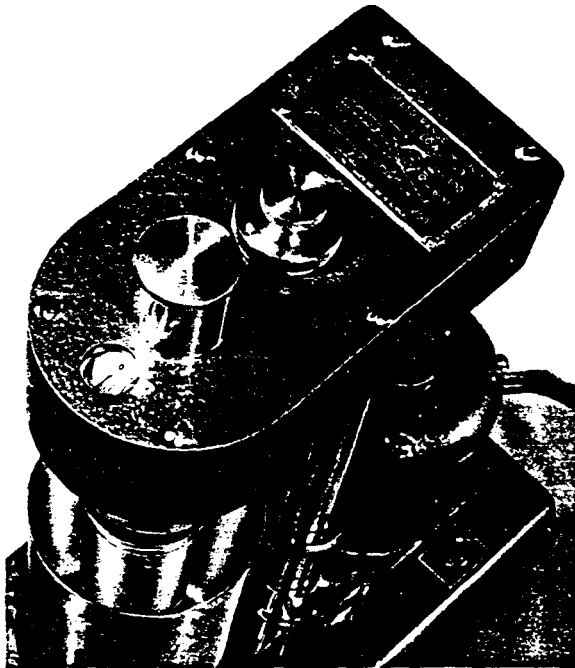
Part No.

- 3466 Hardwood carrying case
- 3466-6 Case with built-in 115-6v transformer
- 3466-12 Case with built-in 115-12v transformer
- 3467 Cord, connecting, 5 ft., Amphenol plug both ends
- 3468 Cord, connecting, 5 ft., Amphenol plug one end, battery clips on other



Model 34 with transformer type carrying case. Steps down 115 volts AC to 6 or 12 volts.





**RHEOLOGICAL ANALYSIS** *with FANN V-G METERS*  
*offers these outstanding advantages:*

Moderate cost.

World-wide distribution.

Flexible basic design to meet any requirement in shear rate level, viscosity range, operational environment.

Rapid torque balancing for fast reading.

Continuous observation of time-dependent properties.

Equilibrium data assured with direct indicating dial.

Known shear rate conditions.

Precision designs for accurate data analysis.

Portable models for 6-12 volt AC/DC operation.

Laboratory models for 115 volt-60 cycle, 115 volt-50 cycle, transformers available for 230 volt systems.

Completely assembled and calibrated prior to shipment.

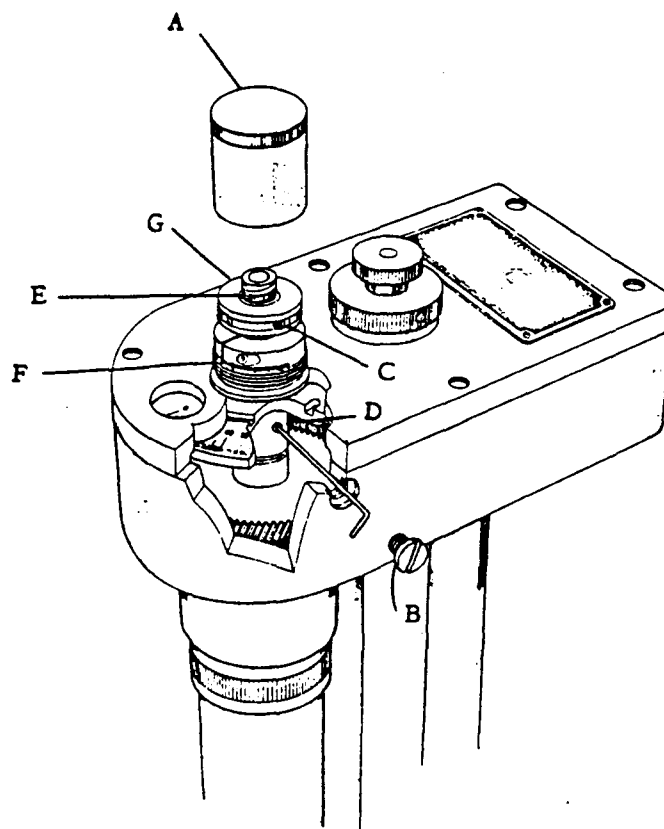
**fann instrument corporation**

P. O. BOX 6101

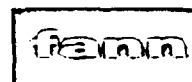
• 3202 ARGONNE ST. •

HOUSTON, TEXAS

# TO CHANGE TORSION SPRINGS IN FANN VISCOMETERS



1. Remove dust cap A and plug screw B.
2. Loosen set screws C and D approximately  $\frac{1}{2}$  turn. Spring can then be lifted out.
3. Insert new spring, making sure that it is properly seated. The top of threaded mandrel inside spring should be exactly flush with clamp ring E. Tighten set screws.
4. Cap screw F can be loosened, permitting knob G to be turned to bring dial "zero" under the index line. Knob G is also movable vertically so that spring can be clamped in a "free" position - neither compressed or stretched.
5. Tighten all screws, replace dust cap and plug screw. For best accuracy check spring constant according to directions given in enclosed spring chart.



**fann instrument corporation**

## OPERATING INSTRUCTIONS FOR MODEL 35 FANN V-G METER

### GENERAL

1. Connect instrument to 115 volt, 60 cycle, A. C. power source.
2. Speeds, rotor sleeve: **CHANGE GEARS ONLY WHEN MOTOR IS RUNNING.**  
600 rpm - Gear shift knob down, motor switch at "high"  
300 rpm - Gear shift knob down, motor switch at "low"  
200 rpm - Gear shift knob up, motor switch at "high"  
100 rpm - Gear shift knob up, motor switch at "low"  
6 rpm - Gear shift knob center, motor switch at "high"  
3 rpm - Gear shift knob center, motor switch at "low"

### PLASTIC VISCOSITY AND YIELD VALUE

1. Place recently agitated sample in test cup furnished. A line scribed inside the cup at 350 cc level is for barrel equivalent volume. With rotor sleeve running at 600 rpm, wait for dial to come to steady value. Note dial reading.
2. Set motor switch at low and obtain 300 rpm reading in the manner outlined in step one.
3. 600 reading - 300 reading equals plastic viscosity (p. v.) in cps.  
300 reading - (p. v.) equals yield value (y. v.) in lbs./100 sq. ft.

### GEL STRENGTH

1. Stir sample thoroughly at 600 rpm.
2. Change gear shift to center then turn motor off.
3. Allow desired rest time (10 seconds to 10 minutes) then turn switch to low (3 rpm).
4. Read dial at instant of gel break. Reading is in lbs./100 sq. ft.

### CONSISTENCY CURVES AND THIXOTROPIC STUDIES

1. For a more complete consistency curve take readings at 200, 100, and 6 rpm in addition to the above. Plot results, speed vs. dial deflection, and extrapolate straight portion of curve to stress axis to obtain yield value intercept.
2. Thixotropic studies are usually made by taking a series of readings beginning at slow speed on unstirred sample. Measurements are made at successively higher speeds to maximum then the downcurve is run immediately. Plotted, the hysteresis loop formed by the upcurve and downcurve is a measure of thixotropy.

Clean instrument by running at high speed with rotor sleeve immersed in water or other solvent. Remove rotor sleeve by twisting slightly to release lock pin. Wipe bob and other parts thoroughly.



**INTERCHANGABLE TORSION SPRINGS  
FOR MODELS 34, 35 and 35S, FANN V-G METERS**

**MAXIMUM MEASURABLE VISCOSITIES - Cps  
(With standard bob and rotor combination)**

(1) For dial reading of 262.3  
use, gms:

		12	30	60	120	180	240	300		
		Standard								
Spring No.	Spring Constant K <sub>s</sub>	27S	25S	25D	22S	21S	22D	20S	20D*	
		77.4	193.5	387	774	1161	1548	1935	3870	
Range Factor		1/5	1/2	1	2	3	4	5	10	
Rotor RPM	600	30	75	150	300	450	600	750	1500	
	300	60	150	300	600	900	1200	1500	3000	
	200	90	225	450	900	1350	1800	2250	4500	
	100	180	450	900	1800	2700	3600	4500	9000	
	6	3000	7500	15,000	30,000	45,000	60,000	75,000	150,000	
	3	6000	15,000	30,000	60,000	90,000	120,000	150,000	300,000	

\*For Use Only with special closed-end rotor

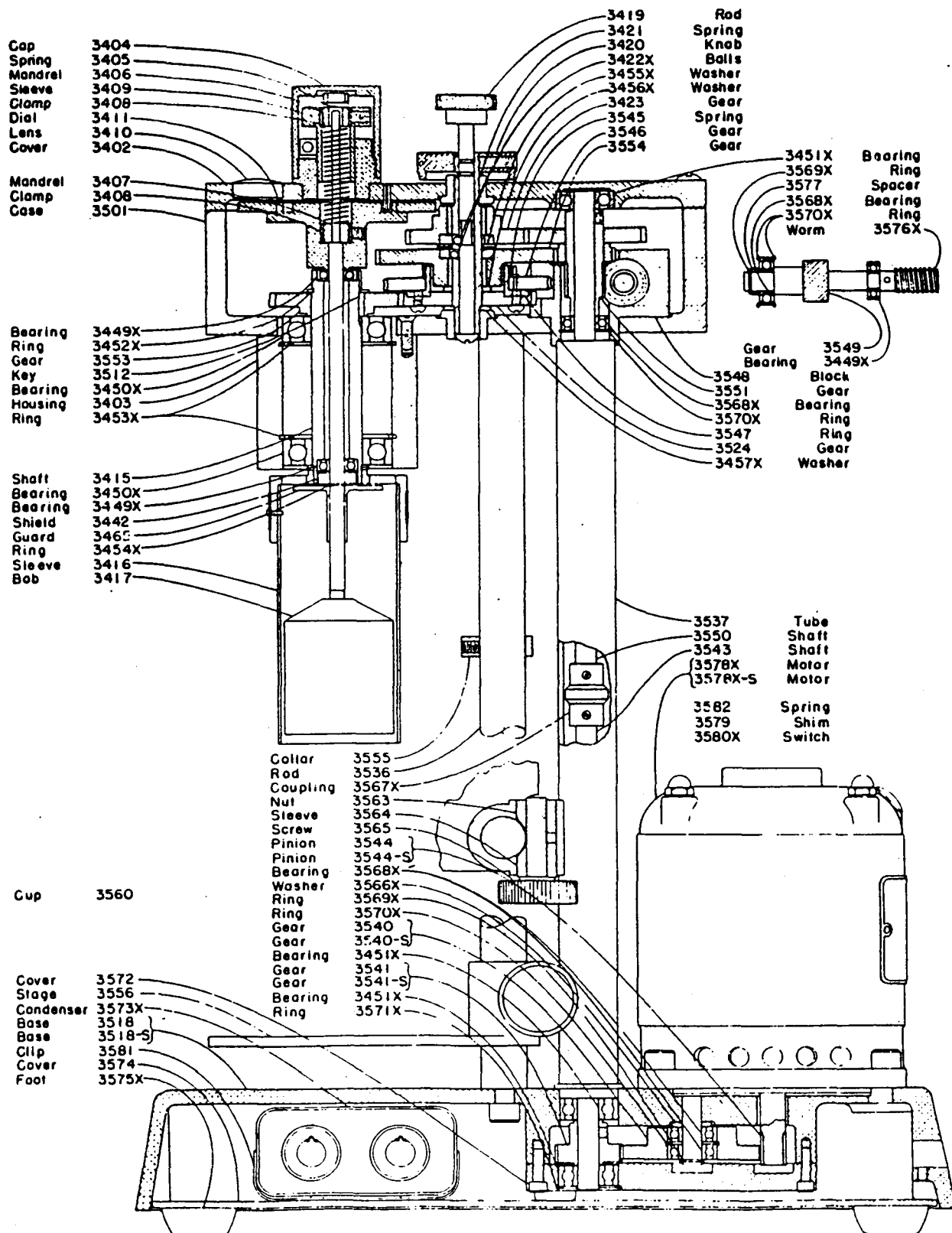
**TO USE:** Install desired spring in instrument and operate in normal manner. Multiply dial reading by range factor.

**NOTE:** All springs are calibrated at the factory but for best accuracy, calibration should be rechecked after installation, by dead weight method or by known fluids.

Formula for calculating K<sub>s</sub>:

$$K_s = \frac{G g R}{\Theta} \quad \text{where:} \quad \begin{array}{l} K_s = \text{Spring Constant - Dyne cm/deg.} \\ G = \text{Load in grams} \\ g = 981 \\ R = \text{Radius arm - cm} \\ \Theta = \text{Dial reading} \end{array}$$

(1) Wrap light string or thread around bob or other cylinder of known diameter fastened to bob shaft. Run string over a pulley and hang weights on string to deflect spring. Adjust free length of spring to get desired deflection.



FANN V-G METER  
MODEL 35

**APPENDIX F**

**TEST PROCEDURE FOR MUD BALANCE "TRU-WATE CUP"**

**(MODIFIED OPERATING INSTRUCTIONS FOR STANDARD TRU-WATE CUP)**

# MUD BALANCE MODIFIED INSTRUCTIONS

## OPERATION

1  
Warning: Blenday (low speed 15  
- full speed - 10 seconds. <sup>secs</sup>

### Operating Instructions

#### TRU-WATE CUP

Part No. 459.047 - Standard

Part No. 459.04900 - Metric

The TRU-WATE Cup (Figures 1 through 4) is an instrument for measuring the absolute density of a fluid sample. The unit is similar in operation to a conventional mud scale, the difference being that the slurry can be placed in a fixed volume sample under pressure.

The purpose of placing the sample under pressure can be explained as follows: A major problem found in the density measurement of fluids, i.e., cement slurries, is that oftentimes these fluids have a considerable amount of air entrained within them. By pressurizing the sample cup the entrained air volume can be decreased to a negligible quantity.

The operation of the TRU-WATE Cup is illustrated in the operational schematics of Figures 5 through 8. The steps involved are outlined here.

#### Step 1

The sample cup initially is filled with the slurry, the density of which is to be found. The cup should be filled to a level slightly below the upper edge of the cup (approximately 1/4").

#### Step 2

Place the lid on the cup with the attached check valve in the down (open) position. Push the lid downward into the mouth of the cup until surface contact is made between the outer skirt of the lid and the upper edge of the cup. Any excess slurry will be expelled through the check valve. When the lid has been placed on the cup, pull the check valve up into the closed position, rinse off the cup and threads with water and then screw the threaded cap onto the cup.

--Prepared by Technical Communications  
For The Engineering Department

## STEP 2B

FOLLOW THE PROCEDURE IN STEP 6  
TO OBTAIN THE DENSITY OF THE  
UNPRESSURIZED SLURRY. IT IS IMPORTANT  
THAT THE MUD BALANCE CUP IS FULL  
FOR THIS STEP, CONTINUE WITH STANDARD  
PROCEDURE TO OBTAIN PRESSURIZED SLURRY  
DENSITY.

**Step 3**

The pressurizing plunger is similar in operation to a syringe. The plunger is filled by submersing the nose of the plunger assembly in the slurry with the piston rod in the completely inward position. The piston rod then is drawn upward, thereby filling the plunger cylinder with slurry.

**Step 4**

The nose of the plunger is pushed onto the mating O-ring surface of the valve. The sample cup is pressurized by maintaining a downward force on the cylinder housing in order to hold the check valve down (open) and at the same time forcing the piston rod inward. Approximately 50 pounds of force or greater should be maintained on the piston rod.

**Step 5**

The check valve in the lid is pressure actuated, which means that when pressure is applied within the cup this same pressure tends to push the valve upward into the closed position. The valve is, therefore, closed by gradually easing up on the cylinder housing while holding pressure on the piston rod. When the check valve closes, disconnect the plunger.

**Step 6**

The pressurized slurry sample now is ready for weighing. The exterior of the cup should be rinsed off and wiped dry. The instrument then should be placed on the knife edge as illustrated. The sliding weight should be moved left or right until the beam is balanced. The beam is balanced when the attached bubble is centered between the two black marks. The density now is obtained by reading one of the four calibrated scales on the arrow side of the sliding weight.

On the standard scale density can be read directly in units of lbs/gal, specific gravity, psi/100 ft., and lbs/cu. ft. On the metric scale, density can be read in units of lbs/gal, specific gravity/kg per liter, kPa/M, and kg/M<sup>3</sup>.

## OPERATION

3

### Step 7

The pressure is released by pushing the valve downward. This is done by reconnecting the empty plunger assembly and pushing downward on the cylinder housing.

The cup should then be emptied of its contents and all components should be thoroughly rinsed and cleaned with water.

For best operation, the valve, lid, and cylinder should be greased frequently with a waterproof grease such as "Lubri-Plate".

### STEP 8

USE THE FOLLOWING EQUATION TO CALCULATE AIR ENTRAINMENT.

$$\text{AIR ENTRAINMENT} = \left( 1 - \frac{\text{MUD BALANCE READING}}{\text{PRESURIZED MUD BALANCE READING}} \right) \times 105.9$$

THIS WILL YIELD AIR ENTRAINMENT IN PERCENT.

TYPICALLY SLURRIES CAN BE HANDLED BY HALLIBURTON'S MIXING EQUIPMENT WITH AIR ENTRAINMENT AS HIGH AS 6 TO 8 %, HOWEVER, AIR ENTRAINMENT ABOVE 8% HAS THE POTENTIAL TO CREATE PUMP CAVITATION THUS MIXING PROBLEMS,

**APPENDIX G**  
**TEST PROCEDURE FOR**  
**QUAMTACHROME MULTIPYCNOMETER OPERATION**



## RF-10 - BULK DENSITY BY MULTIPYCNOMETER

### 1.0 SCOPE

The purpose of this test is to perform bulk densities on pozzolans, a mixture of flyash, lime, and cement, using a multipycnometer. Accurate bulk density measurements are important for maintaining proper feed mixtures.

### 2.0 SUMMARY OF METHOD

The multipycnometer was chosen as an instrument and method for measuring bulk density over conventional weight/volume techniques which involve hydration. Hydrating pozzolans, flyash, and cement partially dissolves the material to be tested yielding inaccurate bulk density determinations. The multipycnometer offers sample volume variability enabling more accurate determinations than if a pycnometer was used. The basic principle of the instrument involves introducing samples into a known volume, purified helium gas is applied at 17-18 PSI pressure which causes a sample volume reduction to occur. A simple calculation yields bulk density results once the reduction in volume is measured. Sample results are available within 1 hour after start of the test.

### 3.0 INTERFERENCES

Many samples contain impurities on their surface and within their pores. The presence of these impurities could cause inaccurate weight measurements or erroneous volume measurements. Volatile organics and inorganic matter can also cause volume displacement error. The presence of volatile contaminants is generally observed by successive volume determinations with results trending in one direction, after each depressurization.

Additional interferences can be caused by high surface area powders in the volume created between the powder surface and the center of the mass of the gas molecules (helium) at the interface. Corrections for this error can be made if Van der Waals diameter of the gas and the powder's specific surface area are known.

### 4.0 PROCEDURE

- 4.1 Follow installation and components for assembly of equipment on pages 3-4 of QUAMTACHROME CORPORATION'S MULTIPYCNOMETER instruction manual for model MVP-1.

## 4.2 Sample Preparation

### 4.2.1 Purging

To purge contamination gas or vapor from the system attach a length of hose to the "VENT" hose connection on the right side of the MULTIPYCNOMETER and immerse the other end of the hose into a beaker of water. Then perform the following steps.

- 4.2.1.1 Close the "GAS IN" toggle valve and open the "GAS OUT" toggle valve. Turn selector valve to "CELL".
- 4.2.1.2 Open "GAS OUT RATE" control fully counter-clockwise.
- 4.2.1.3 Close "GAS IN RATE" needle valve fully clockwise. Do not force or overtighten. Open the "GAS IN" toggle valve.
- 4.2.1.4 Adjust "GAS IN RATE" needle valve to give a slow rate of bubbling in the beaker of water, then remove tubing from the water.
- 4.2.1.5 After 10-20 minutes of flow, close the "GAS IN" toggle valve.
- 4.2.1.6 See Section IV of the manufacturer's manual for sample volume measurement steps.

### 4.2.2 Vacuum

If it is necessary to decontaminate the powder with vacuum use the following procedure.

- 4.2.2.1 Close the "GAS IN" toggle valve and open the "GAS OUT" toggle valve with the Selector valve turned to "CELL". Close "GAS OUT RATE" fully clockwise. Do not overtighten.
- 4.2.2.2 Attach vacuum tubing to the "VENT" hose connection.
- 4.2.2.3 Very slowly open the "GAS OUT RATE" control. If the "GAS OUT RATE" control is opened too rapidly, powder can be pulled out of the cell holder.

- 4.2.2.4 To repressurize the system, close the "GAS OUT RATE" control and remove vacuum tubing. Open the "GAS IN" toggle valve. Slowly open the "GAS IN RATE" control valve to admit helium gas until the system is returned to ambient pressure.

### 4.3 Analyzing Samples

If, for any reason, it is suspected that the values of  $V_c$  or  $V_a$  has been altered then recalibrate the unit. Calibration should be performed if powder blows out of the sample cell into the tubing, or operating at substantially different than room temperature. Refer to earlier referenced operational manual section V, Calibration pages 7-10. Refer to Figure 1 for instrument drawing.

- 4.3.1 Turn power "ON", allow 10-15 minutes for the pressure transducer to warm up and stabilized.
- 4.3.2 Select the correct REFERENCE VOLUME for the sample cell to be used. SEE THE FRONT PANEL FOR REFERENCE VOLUME SELECTION.
- 4.3.3 Weigh the empty sample cup to nearest 0.01 g and record the weight in the logbook.
- 4.3.4 Fill the sample cup and weigh to nearest 0.01 g and record the weight in the logbook; then insert the sample cup into the cell holder and replace the cover.
- 4.3.5 Open the "GAS OUT" and "GAS OUT RATE" control. Wait for a stable zero reading.
- 4.3.6 Close the "GAS OUT" toggle valve and set the meter to zero. If unable to zero the display, center the zero control knob, remove the rear panel and adjust the  $R_1$  variable resistance on the printed circuit board.
- 4.3.7 Turn the selector valve to "REF".
- 4.3.8 Open the "GAS IN" toggle valve, and pressurize to approximately 17 PSIG (1.195Kg/cm<sup>2</sup>) using the "GAS IN RATE" needle valve to control the rate of pressurization. Stop the flow by closing the "GAS IN" toggle valve.

CLOSE  
GAS OUT RATE CONTROL

- 4.3.9 Record the display reading after is has stabilized. This value is " $P_1$ ".
- 4.3.10 Turn the selector valve to "CELL".
- 4.3.11 Record the display reading after is has stabilized. This value is " $P_2$ ".
- 4.3.12 Vent the pressure slowly to prevent blowing powder out of the cell, by opening the "GAS OUT" toggle valve with the "GAS OUT RATE" control slightly open.

**Note:** The pressure transducer used in this pycnometer dissipates a very slight amount of heat. Because of its extreme sensitivity it can track the slight pressure increases associated with the heating of the gas. Accordingly, it is necessary to take the first reading observed after the digital display stabilizes. A change of approximately 0.001 on the digital display every 10-20 seconds is indicative of pressure increases due to heat dissipation and is normal.

## 5.0 CALCULATIONS

The true volume of powder is calculated by the following equation:

$$V_p = V_c - V_r ((P_1/P_2)) - 1)$$

Where:

$V_p$  = Volume of powder in  $\text{cm}^3$

$V_c$  = Volume of sample cell in  $\text{cm}^3$

$V_r$  = Reference volume in  $\text{cm}^3$

$P_1$  = Pressure reading after pressurizing referenced volume.

$P_2$  = Pressure reading after including  $V_c$

Density is calculated by the following equation

$$\text{Density g/cm}^3 = \frac{\text{Weight of sample in grams}}{V_p \text{ in cm}^3}$$

## 6.0 REAGENTS

Purified 99.999% helium.

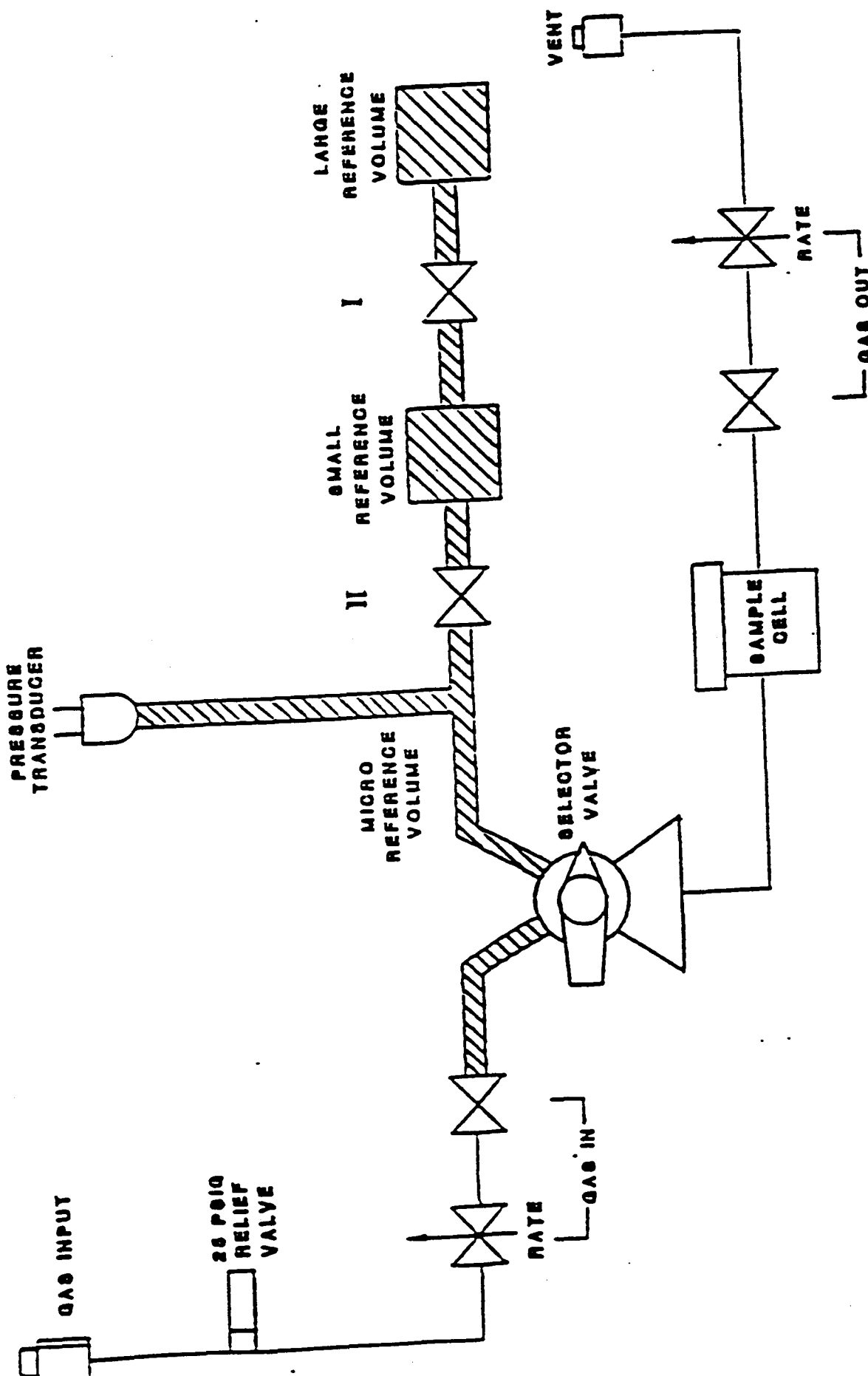
## 7.0 APPARATUS

- Multipycnometer
- Analytical balance
- Two stage gas regulators

## 8.0 REFERENCE

QUAMTACHROME CORPORATION MULTIPYCNOMETER instruction manual,  
10/91, for model MVP-1.

Figure 1 - MULTIPYCNOMETER



## V. CALIBRATION

The first page of this manual contains calibration information. This includes the sample cell ( $V_c$ ), the reference volumes ( $V_R$ ), the volume of the large calibration sphere ( $V_{cal, large}$ ) and the volume of the small calibration spheres ( $V_{cal, small}$ ) provided with the MULTIPYCNOMETER. If, for any reason, it is suspected that the value of  $V_c$  or  $V_R$  has been altered then recalibration should be performed. Powder blowing out of the sample cell into the tubing or operation at temperatures significantly different than room temperature will require recalibration.

All reference and cell volume calibrations are traceable to the independently calibrated spheres used with the instrument. To recalibrate the MULTIPYCNOMETER the following steps should be followed in order.

1. Insert the large cell into the cell holder. Place the calibration sphere into the large cell holder and close the cover.
2. Open valves I and II.
3. Turn the selector valve to "CELL".
4. Open the "GAS OUT" toggle and "RATE" valves.
5. Open the "GAS IN" toggle valve and adjust the "RATE" valve until the display shows 17 PSI.
6. Purge the MULTIPYCNOMETER in this mode for about 5 minutes.
7. Close the "GAS IN" toggle valve.
8. When the display shows a stable reading, set it to zero using the zero control knob and turn the selector valve to "REF".
9. Close the "GAS OUT" toggle valve.
10. Open the "GAS IN" toggle valve until the pressure is approximately 17 PSI. Then close the "GAS IN" toggle valve.
11. When the display is stable, note the pressure reading.
12. Turn the selector valve to "CELL".

13. When the display is again stable, note the new pressure reading.
14. Vent the MULTIPYCNOMETER by opening the "GAS OUT" toggle valve.
15. Remove the calibration sphere from the large cell and repeat steps 1-14 noting the pressure with the selector valve in "REF" and "CELL" positions.
16. Calculate the volume of the large reference Volume ( $V_{R \text{ large}}$ ) using equation.

$$V_{R \text{ large}} = \frac{V_{\text{cal}}}{\left[ \left( P_1' \text{ large} / P_2' \text{ large} \right) - 1 \right] - \left[ \left( P_1 \text{ large} / P_2 \text{ large} \right) - 1 \right]}$$

Where:

$V_{\text{cal}}$  = volume of the calibration sphere

$P_1' \text{ large}$  = pressure in  $V_{R \text{ large}}$  with no sphere in the cell.

$P_2' \text{ large}$  = pressure in  $V_{R \text{ large}}$  and the large cell with no sphere in the cell.

$P_1 \text{ large}$  = pressure in  $V_{R \text{ large}}$  with the calibration sphere in the cell.

$P_2 \text{ large}$  = pressure in  $V_{R \text{ large}}$  and the large cell with the sphere in the cell.

17. After solving equation (9) for  $V_{R \text{ large}}$  use this value in equation (10) to calculate  $V_{C \text{ large}}$  the large sample cell.

$$V_{C \text{ large}} = V_{\text{cal}} + V_{R \text{ large}} \left( \left( P_1 \text{ large} / P_2 \text{ large} \right) - 1 \right) \quad (10)$$

18. Close toggle valve 1 and repeat steps 8-14.
19. Calculate the small reference volume using equation 11.

$$V_{R \text{ small}} = \frac{V_{C \text{ large}}}{\left[ \left( P_1 \text{ small} / P_2 \text{ small} \right) - 1 \right]}$$



Where:

$P_1$  = pressure in  $V_R$  with no sphere in the cell.

$P_2$  = pressure in  $V_R$  and the large cell with no sphere in the cell.

20. Remove the large sample cell and insert the small adapter sleeve and small sample cell.
21. Repeat steps 3-14 and calculate the small cell volume using equation 12.

$$V_{c\text{small}} = V_{R\text{small}} \left[ \left( \frac{P_1' \text{small}}{P_2' \text{small}} \right) - 1 \right]$$

Where:

$P_1' \text{small}$  = pressure in  $V_{R\text{small}}$  with no sphere in the cell.

$P_2' \text{small}$  = pressure in  $V_{R\text{small}}$  and the small cell with no sphere in the cell.

22. Remove the small sample cell and adapter and insert the micro adapter sleeve, micro cell and two small calibration spheres.
23. Close toggle valves I and II.
24. Open the "GAS IN" toggle valve until the pressure is approximately 17 PSI. Then close the "GAS IN" toggle valve.
25. When the display is stable, note the pressure reading.
26. Turn the selector valve to "CELL".
27. When the display is again stable, note the new pressure reading.
28. Vent the MULTIPYCNOMETER by opening the "GAS OUT" toggle valve.
29. Remove the calibration spheres from the micro cell and repeat steps 24-28 noting pressures with the selector valve in "REF" and "CELL" positions.
30. Calculate the volume of the micro reference volume ( $V_{R\text{micro}}$ ) using equation (13).

$$V_{R\text{ micro}} = \frac{V_{\text{cal}}}{\left[ (P_1' \text{ micro} / P_2' \text{ micro}) - 1 \right] - \left[ (P_1 \text{ micro} / P_2 \text{ micro}) - 1 \right]}$$

Where:

$V_{\text{cal}}$  = volume of the calibration spheres

$P_1' \text{ micro}$  = pressure in  $V_{R\text{ micro}}$  with no spheres in the cell

$P_2' \text{ micro}$  = pressure in  $V_{R\text{ micro}}$  and the micro cell with no spheres in the cell.

$P_1 \text{ micro}$  = pressure in  $V_{R\text{ micro}}$  with the calibration spheres in the cell.

$P_2 \text{ micro}$  = pressure in  $V_{R\text{ micro}}$  and the micro cell with the spheres in the cell.

31. After solving equation (13) for  $V_{R\text{ micro}}$  use this value in equation (14) to calculate  $V_{C\text{ micro}}$  for the micro sample cell.

$$V_{C\text{ micro}} = V_{\text{cal}} + V_{R\text{ micro}} \left( (P_1 \text{ micro} / P_2 \text{ micro}) - 1 \right)$$

NOTE: If only the large sample cell is to be used, it is not necessary to recalibrate the small and micro cells. Similarly, if only the small cell is to be used, it is not necessary to calibrate the micro cell.

If the sample is analyzed at a temperature different by more than 4°C from that used to calibrate the instrument, the MULTIPYCNOMETER should be recalibrated at the new temperature.

**APPENDIX H**

**CONCEPTUAL OUTLINE OF PONDCRETE AND  
SALTCRETE TREATABILITY STUDY**

**MEMORANDUM**

**MARK SPERANZA TO TED BITTNER - REVISION 1**



## INTERNAL CORRESPONDENCE

C-49-12-2-154

**TO: TED BITTNER**

**DATE: DECEMBER 18, 1992**

**FROM: MARK SPERANZA**

**cc: R. NINESTEEL**

**SUBJECT: CONCEPTUAL OUTLINE OF PONDCRETE  
AND SALTCRETE TREATABILITY STUDY  
REVISION 1**

**T. SNARE**

**R. SIMCIK**

**J. SCHMIDT**

**J. ZAK**

**File: 2K68.231**

The Treatability Study will consist of three phases. Phase I will include physical testing and other testing necessary to determine major process options, thus allowing earlier equipment selection. Phase II will consist of testing to screen various cement stabilization and solidification (CSS) formulas for key parameters, including additives for TCLP compliance. Phase III will consist of testing to determine appropriate operating ranges for key parameters and to demonstrate regulatory compliance. Phase III will also consist of additional studies for miscellaneous concerns such as superplasticizer addition.

MPS/pam

## CONCEPTUAL OUTLINE OF PONDCRETE TREATABILITY STUDY

### PONDCRETE PHASE I

This Phase will consist of two subparts, IA and IB. Phase IA will consist of tests to determine the upper limit of the waste loading, and engineering parameters such as viscosity, bulk densities, specific gravities, etc. Phase IB will consist of dewatering studies and trash studies. Additional details are provided below.

#### **Pondcrete Phase IA**

##### Initial Analytical Testing of Waste Feed

#### 1. Methanol Study for Triwalls in Metal Containers:

- a. Conduct total methanol analysis with second column confirmation to verify presence of contaminant for triwalls in metal containers.
- b. Conduct zero head space TCLP extraction for methanol. Conduct analysis using a spike sample at the average and 2X the average concentration of methanol as determined by the characterization study.

Goal: The goal of this study is to determine whether methanol will leach above the LDR standard in its existing state (waste feed).

#### 2. Baseline Analysis:

- a. Both triwalls and the triwalls in the metal containers will be analyzed for TCLP metals, total metals, anions and cations.

Goal: Acquire data for the bulk samples (waste feed material to compare with characterization data.

3. Engineering Parameters:

- a. Analyze triwalls and triwalls in metal containers for bulk density, specific gravity of the discrete particles, moisture content, and Karl Fisher percent water.

Goal: Determine baseline for engineering parameters of the waste feed.

Engineering Studies

1. Waste Loading Study: This study will be conducted for triwalls and triwalls in metal containers. The study will consist of mixing the waste (feed) forms, at different percent solids, with the same CSS formulation used for the solar ponds. The CSS formulation will consist of portland Type V cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.075, respectively. The waste (feed) loading will vary based on the total solids content of the waste. Mixes will be conducted at 70, 60, 50, 40, 30, and 20 percent solids. The saturated liquid phase of the slurry (concentration determined by the dissolution test) will also be solidified using the same CSS formulation. All mixes will be prepared at a water-to-pozzolan ratio of 0.42. Trash inclusion will not be tested in this phase, but will be evaluated in Phase IB values unless otherwise noted. Testing of the input and output waste streams will be as follows:

Input (feed waste): Total solids, total dissolved solids of supernatant, viscosity, specific gravity, and density.

Output (product slurry): Density, VG Fann testing, and observation of the ability of the material to be pumped (the testing will be filmed).

Testing: The product slurry will be placed in cylinders to be cured using the 48-hour warm water accelerated cure procedure. Cylinders will be submitted for 48-hour UCS, TCLP metal analysis, and accelerated durability testing. All cylinders will be evaluated for free liquid after curing by visual observation.

Goal: This testing will determine the upper limit of the waste loading, which will help determine the degree of dewatering which will be necessary (e.g., if 20 percent was the upper waste loading, then it would not be necessary to dewater the waste material to greater than 20 percent solids). The results of this testing should narrow the selection of downstream process options.

2. Dissolution Test of Pondcrete in Water: This test consists of dissolving a known mass of waste feed sample in excess water and determining the specific gravity and TDS of the solution. The dry weight of the undissolved portion of the sample will also be determined. The undissolved portion of the sample will be analyzed for specific gravity of the discrete particles.

Another test will consist of dissolving the sample in a small amount of water to make a saturated solution. The TDS and specific gravity of the supernatant will be determined after filtration.

Both these tests will be performed at room temperature and repeated at 100°F.

3. **Viscosities and Densities of Samples at Different Percent Solids:** Mixtures of the waste feed samples with water will be prepared at different total solids concentrations (from 5% to 60%). The viscosities and specific gravities will be determined for each of the samples. The viscosities of the supernatant, up to the saturated point, will also be determined. The experiment will be conducted at room temperature and at 100°F to reflect the expected temperature rise from grinding.
4. **Settling Tests:** Settling tests will be conducted on waste feed samples ground to -10 mesh to determine settling rates and terminal densities. These tests will use a saturated solution for the liquid phase and be conducted at different percent solids and temperatures.
5. **Rheology Evaluation of Slurries:** Viscosity of the waste feed will be measured over a range of solids (5 to 50 percent) with and without trash included in the slurry. This testing will be conducted in a saturated solution. Trash is defined as the pallet, plastic sheeting, and steel band. The component "steel band" will include iron straps, nails, and iron ball shavings from the grinding process. The trash makes up approximately 8% of the total weight of the billet on a wet weight basis. When using dry weights, 20% of the billet is considered to be trash. Specific gravities of the slurries will also be measured.
6. **Saturation TDS Versus Temperature:** The degree of salt dissolution versus temperature will be determined by collecting samples of the supernatant at different temperatures (i.e., 50°F, room temperature and 100°F). The samples will be analyzed for TDS and specific gravity.



## **Pondcrete Phase IB**

This portion of Phase I will consist of evaluating dewatering processes and performing a trash study. The dewatering study will evaluate the selected process option which is believed to be the most practical to achieve the percent solids as determined in the waste loading study. The trash study will evaluate various loadings of trash to determine if there is any impact from different loadings.

1. Dewatering Studies: This testing will evaluate various dewatering processes which are capable of achieving the appropriate percent solids of the waste feed which will be determined in the waste loading study. Vendors will be solicited to conduct the dewatering tests. The dewatering testing will include the appropriate quantities of trash. Further details on these tests will be determined at the completion of the waste loading test.
2. Trash Study: This study will evaluate trash addition with regards to physical parameters and chemical parameters. Testing will be conducted at the percent solids determined to be the maximum waste loading. Trash will be added at 2, 5, 7.5, 10, 12.5, 15, 20, 50, 75, and 100 percent of the waste feed loading. The objective of this test is to generate data to eliminate trash as a future variable for the CSS formulation development.
  - a. Physical testing: Trash will be blended with the triwalls and the triwalls in the metal containers. Testing of waste feed will consist of the following:
    - Percent solids
    - Bulk density
    - Viscosities

- Specific gravities
  - Karl Fisher
- b. CSS Testing: CSS testing of the product slurry will be conducted to determine the effect of trash on TCLP criteria and on the stability of the solidified product. The CSS testing will be conducted at three waste loadings; one will be the selected waste loading from the initial waste loading study and the other two will bracket either side of the selected loading (e.g., 40, 50, and 60 percent solids). The water-to-pozzolan ratio will be at 0.42. The product slurry will be placed in plastic cylinders to be cured using the 48-hour accelerated cure method followed by UCS, TCLP metal analysis, accelerated durability testing, and visual observation for free liquids.

#### **PONDCRETE PHASE II - CSS FORMULATION DEVELOPMENT**

This Phase will be conducted using triwalls. Triwalls in metal containers will not be used unless concerns with methanol still exist. Triwalls have the same contaminants as those that exceeded the LDRs for the triwalls in metal containers but at higher concentrations. These studies will be conducted at the selected waste loading using water-to-pozzolan ratios of 0.34, 0.42, and 0.50. The CSS formula will consist of Type V cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.075. This testing will determine the need for additives to improve the characteristics of the solidified product so that it will pass the TCLP testing criteria.

The first set of tests will be cured for 48 hours and then submitted for TCLP metal analysis. Depending on the results of these test, further testing may be required to evaluate various additives to reduce the leachability of the metal constituents. If the TCLP results pass the LDR criteria then this Phase will be

complete.

**NOTE:** Testing in this phase will be dependent on the results of the waste loading study. Upon completion of the waste loading study, additional information will be available to thoroughly scope the CSS formulation development.

### **PONDCRETE PHASE III - REGULATORY TESTING PHASE**

This Phase will test the CSS formulation to determine if the product will pass all regulatory criteria over the proposed operating range. The proposed operating range will be at a water-to-pozzolan ratio of 0.34 to 0.50 with 0.42 being the center point. These water-to-pozzolan ratios will be tested at +/-10 percentage points around the selected waste loading. The test cylinders, produced from the product slurry, will be cured for 7 days and 28 days. After 7 days the cylinders will be tested for TCLP metals and UCS. After 28 days the cylinders will be tested for TCLP metals, UCS, paint filter liquids, liquids/solids, and durability testing.

Several additional tests will also be conducted during this Phase of the work. A factorial experiment will be conducted which varies the ratio of pozzolans so that the cement to flyash to lime ratio can be varied from 1/2/0.075 during remediation. Another factorial experiment will be conducted for superplasticizer addition. The test cylinders will also be cured at different temperatures to evaluate the heat generated during full scale curing. Cylinders from each of these three tests will be tested for all of the pertinent regulatory criteria.

## **CONCEPTUAL OUTLINE OF SALTCRETE TREATABILITY STUDY**

### **SALTCRETE PHASE I**

This Phase will consist of two subparts, IA and IB. Phase IA will consist of tests to determine the upper limit of the waste loading, and engineering parameters such as viscosity, bulk densities, specific gravities, etc. Phase IB will consist of dewatering studies and trash studies. Additional details are provided below.

#### **Saltcrete Phase IA**

##### **Initial Analytical Testing of Waste Feed**

##### **1. Total dissolved solids test:**

This test will be conducted with the triwalls, half crates, and triwalls in metal containers. Each waste feed form will be dissolved in excess deionized water at different dilutions. The liquid will then be filtered and the filtrate will be analyzed for TDS. The filter cake will be analyzed for TS and specific gravity.

Goal: To provide an initial baseline of the salt in each of the waste forms.

##### **2. Baseline Analysis:**

Triwalls, half crates, and the triwalls in the metal containers will be analyzed for TCLP metals, total metals, anions, and cations.

Goal: Acquire baseline data for the bulk samples (waste feed material) and compare it with characterization data.

### 3. Engineering Parameters:

Analyze triwalls, half crates, and triwalls in metal containers for bulk density, specific gravity of the discrete particles, moisture content, and Karl Fisher percent water.

Goal: Determine baseline for engineering parameters of the waste feed.

#### Engineering Studies

1. Waste Loading Study: This study will be conducted for triwalls, half crates, and triwalls in metal containers. The study will consist of mixing the waste feed forms, at different solids concentrations, with the same CSS formulation used for the solar ponds. The CSS formulation will consist of portland Type V cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.075, respectively. Batches will also be prepared using lime/cement/flyash and latex. Latex will be evaluated at 5 and 10 percent of the weight of the cement. Mixes will be conducted at 70, 60, 50, 40, 30, and 20 percent total solids of waste feed material. The saturated supernatant will also be solidified using the same CSS formula. All mixes will be prepared at a water-to-pozzolan ratio of 0.42. A corresponding TDS value will also be determined for each waste loading. Trash inclusion will not be tested in this phase but will be evaluated in Phase IB. Testing of the input and output waste streams will be as follows:

Input (waste feed): Total solids, total dissolved solids of the supernatant, viscosity, specific gravity, and density.

Output (product slurry): Density, VG Fann testing, and observation of the ability of the material to be pumped (the testing will be filmed).

Testing: The product slurry will be placed in cylinders to be cured using the 48-hour warm water accelerated cure procedure.

Cylinders will be submitted for 48-hour UCS, TCLP metal analysis, and accelerated durability testing. All cylinders will be evaluated for free liquid after curing by visual observation.

Goal: This testing will determine the upper limit of the waste loading which will determine the degree of dewatering, if any, which will be necessary (i.e., if 50 percent was the upper waste loading, then it would not be necessary to dewater the waste material to greater than 50 percent solids). The results of this testing should narrow the selection of downstream process options. Additionally, this testing will determine whether latex is beneficial and should be considered further during the CSS formulation development.

2. Dissolution Test of Saltcrete in Water: This test consists of dissolving a known mass of waste feed sample in excess water and determining the specific gravity and TDS of the solution. The dry weight of the undissolved portion of the sample will also be determined. The undissolved portion of the sample will be analyzed for specific gravity of the discrete particles.

Another test will consist of dissolving the sample in a small amount of water to make a saturated solution and the TDS and specific gravity of the supernatant determined after filtration.

Both these tests will be performed at room temperature and repeated at 100°F.

3. Viscosities and Densities of Samples at Different Percent Solids: Mixtures of the waste feed samples with water will be prepared at different total solids concentrations (from 5% to 60%). The viscosities and specific gravities will be determined for each of the samples. The viscosities of the supernatant, up to the saturated point, will also be determined. The experiment will be conducted at room temperature and at 100°F to reflect the expected temperature rise from grinding.
4. Settling Tests: Settling tests will be conducted on waste feed samples ground to -10 mesh to determine settling rates and terminal densities. These tests will use a saturated solution for the liquid phase and be conducted at different percent solids and temperatures.
5. Rheology Evaluation of Slurries: Viscosity will be measured over a range of solids (5 to 50 percent) with and without trash included in the slurry. This testing will be conducted in a saturated solution. Trash is defined as the pallet, plastic sheeting, and steel band. The component "steel band" includes: iron straps, nails, and iron ball shavings from the grinding operation. The trash makes up approximately 8% of the total weight of the billet on a wet weight. When using dry weights, 15% of the billet is considered to be trash. Specific gravities of the slurries will also be measured.
6. Saturation TDS Versus Temperature: The degree of salt dissolution versus temperature will be determined by collecting samples of the supernatant at different temperatures (i.e., 50°F, room temperature, and 100°F). Samples will be analyzed for TDS and specific gravity.

## **Saltcrete Phase IB**

This portion of Phase I will consist of evaluating dewatering processes and conducting a trash study. The dewatering study will evaluate the selected process option which is believed to be the most practical to achieve the target percent solids as determined in the waste loading study. The trash study will evaluate various loadings of trash to determine if there is any impact from different loadings.

1. Dewatering Studies: This testing will evaluate various dewatering processes which are capable of achieving the appropriate percent solids of the waste feed, determined in the waste loading study. Vendors will be solicited to conduct the dewatering tests. The dewatering testing will include the appropriate quantities of trash. Further details on these tests will be determined at the completion of the waste loading test.
2. Trash Study: This study will evaluate trash addition with regards to physical parameters and chemical parameters. Testing will be conducted of the TDS concentration determined in the waste loading study. Trash added at 2, 5, 7.5, 10, 12.5, 15, 20, 50, 75, and 100 percent of the waste feed loading. The objective of this test is to generate data to eliminate trash as a future variable for the CSS formulation development.
  - a. Physical testing: Trash will be blended with the triwalls, half crates, and the triwalls in the metal containers. Testing of the waste feed will consist of the following:
    - Percent Solids
    - Percent TDS



- Bulk Density
  - Viscosities
  - Specific Gravities
  - Karl Fisher
- b. CSS Testing: CSS testing of the product slurry will be conducted to determine the effect of trash on TCLP criteria and on the stability of the solidified product. The CSS testing will be conducted at three waste loadings; one will be the selected waste loading from the initial waste loading study and the other two will bracket either side of the selected loading (e.g., 40, 50, and 60 percent solids). The water-to-pozzolan ratio will be at 0.42. The product slurry will be placed in plastic cylinders to be cured using the 48-hour accelerated cure method followed by UCS, TCLP metal analysis, accelerated durability testing, and visual observation for free liquids.

#### **SALTCRETE PHASE II - CSS FORMULATION DEVELOPMENT**

This Phase will be conducted using triwalls, triwalls in metal containers, and half crates. These studies will be conducted at the selected waste loading using water-to-pozzolan ratios of 0.34, 0.42, and 0.50 for the triwalls in metal containers. The half crates and triwalls will be evaluated using higher water-to-pozzolan ratios because treatment of the chemical constituents is not required (neither population exceeded any LDR standards). Using a higher water-to-pozzolan ratio will reduce the volume of the output. A water-to-pozzolan ratio of 1.0 will be evaluated to determine if a stable waste can be produced. Testing for triwalls and half crates will also include accelerated durability to determine the effect of using higher water-to-pozzolan ratios on long-term durability. The CSS formula will consist of Type V cement, Type C flyash, and hydrated lime at a ratio of 1.0/2.0/0.075. Latex will be evaluated if it was determined to be

warranted in the Phase I waste loading study. The testing for triwalls in metal containers will determine the need for additives to improve the characteristics of the solidified product so that it will pass the TCLP testing criteria.

The first set of tests will be cured for 48 hours and then submitted for TCLP metal analysis. Depending on the results of these test, further testing may be required to evaluate various additives to reduce the leachability of the metal constituents. If the TCLP results pass the LDR criteria, then this Phase will be complete.

**NOTE:** Testing in this phase will be dependent on the results of the waste loading study. Upon completion of the waste loading study, additional information will be available to thoroughly scope the CSS formulation development.

#### **SALTCRETE PHASE III - REGULATORY TESTING PHASE**

This Phase will test the CSS formulation to determine if the product will pass all regulatory criteria over the proposed operating range. The proposed operating range for metal containers will be at a water-to-pozzolan ratio of 0.34 to 0.50 with 0.42 being the center point. The operating range for triwalls and half crates will be determined in Phase II. These water-to-pozzolan ratios will be tested at +/-10 percentage points around the selected waste loading. The test cylinders, produced from the product slurry, will be cured for 7 days and 28 days. After 7 days the cylinders will be tested for TCLP metals and UCS. After 28 days the cylinders will be tested for TCLP metals, UCS, paint filter liquids, liquids/solids, and durability testing.

Several additional tests will also be conducted during this Phase of the work. A factorial experiment will be conducted which varies the ratio of pozzolans so that the cement to flyash to lime can be varied from 1/2/0.075 during remediation. Another factorial

experiment will be conducted for superplasticizer addition. Curing the test cylinders at different temperatures will also be evaluated. Cylinders from each of these three tests will be tested for all of the pertinent regulatory criteria.

**APPENDIX I**

**DISPOSAL OF LABORATORY WASTES**

- **MEMORANDUM "WASTEWATER DISPOSAL"**  
**PAUL FRANK TO DON FERRIER**
- **AP-017 "PACKAGING AND SHIPMENT**  
**OF SOLAR POND PROJECT SAMPLES**  
**FOR RETURN TO ROCKY FLATS"**

December 13, 1991

C-28-91-PVF-1052

Mr. Dan Ferrier  
EG&G Rocky Flats

SUBJECT: Wastewater Disposal

Dear Mr. Ferrier:

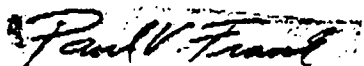
As you know, the contract with EG&G governing our treatability studies requires that HALLIBURTON NUS return to EG&G all residuals, unused sample materials, and wastes from these studies. It has become clear that the wastes generated during the treatability phase of the Rocky Flats Solidification Project will include very substantial volumes of very slightly contaminated water resulting from the cleanup of laboratory equipment, glassware, etc. We are concerned that, apart from the expense of packing and trucking large volumes of such water, its return as liquid offers the unnecessary potential for accidental release and the consequent adverse public reaction, even though no public risk would be created.

Accordingly, HNUS would like to propose a more cost-effective and risk-averse alternative to the return to Rocky Flats of this water, estimated to exceed 50 gallons per day over the 180 days of the treatability phase. This alternative would discharge to the sanitary sewer only that wastewater meeting EPA Drinking Water Standards. Any wastewater not meeting those standards would be sequentially processed with particulate filtration, mixed-bed ion exchange, and charcoal filtration to remove particulates, soluble cations and anions, and any residual organic contaminants. The process media, i.e., the particulate and charcoal filters and IX resins, would be appropriately packaged and returned to EG&G.

The processed wastewater would be analyzed for gross alpha and gross beta activities. If these analyses indicate that the processed wastewater complies with the EPA's Drinking Water Standard (5 pCi  $\alpha$ /L, 50 pCi  $\beta$ /L), it would be discharged to the sanitary sewer system, consistent with the HNUS license from NRC. Processed waste water that does not meet this standard would be packaged and returned to EG&G.

We hope you will find this proposal acceptable, and would appreciate hearing from you as soon as possible.

Sincerely,



Paul V. Frank  
Project Manager  
Laboratory Services Group

PVF/ckh

Copies: Ted Bittner  
Mort Goldman  
Dave Kohl  
Rich Ninesteel

John Schmidt  
Joanne Simanic  
Dave Yesso

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## PACKAGING AND SHIPMENT OF SOLAR POND PROJECT SAMPLES FOR RETURN TO ROCKY FLATS

### 1.0 PURPOSE

This procedure establishes requirements for the packaging of solar pond project samples and their return to the Rocky Flats Plant. The procedure is specific for the solar pond project, and cannot be used for other projects without appropriate modifications.

### 2.0 DEFINITIONS

2.1 Matrices: The following are considered separate matrices for the purposes of this procedure:

- o 207A pond sludge
- o 207B pond sludge
- o 207C pond sludge
- o solidified material from treatability studies
- o pondcrete
- o saltcrete
- o clarifier sludge

2.2 RAM: Used as an abbreviation for radioactive materials.

2.3 LSG: Used as an abbreviation for the Halliburton NUS Environmental Corporation Laboratory Services Group.

### 3.0 RESPONSIBILITIES

#### 3.1 LSG RAM Coordinator

- o Select carrier and make arrangements for each shipment.
- o Contact EG&G Rocky Flats to arrange for receipt of each shipment.
- o Pack drums for shipment. Alternatively, train other LSG personnel in this procedure and supervise packing of the drums.

-----  
Approvals:

Lab Manager	Date	Rad Safety Off. Date	QA Manager	Date
-------------	------	----------------------	------------	------

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- o Prepare the following documents for each shipment:
  - LSG radioactive material shipment record (Figure 8)
  - LSG solar pond project packing slip (Figure 9)
  - LSG chain of custody record (Figure 10)
- o Supervise loading of the trailer bed for each shipment.
- o Placard the truck for each shipment.
- o Follow-up with EG&G Rocky Flats, Inc. contact to obtain return receipt.

### 3.2 LSG Quality Assurance Inspector

- o Inspect each drum prepared for shipment. Apply chain of custody seal(s) to the drum following successful inspection.
- o Inspect the trailer bed following loading. Apply chain of custody seal to the trailer door following successful inspection.
- o Document inspections on the inspection checklist. (Figure 7)

## 4.0 PROCEDURE

Solid and semisolid sample residuals are placed into paint cans or plastic pails. These are placed into large, steel drums. Liquid samples, received from the Rocky Flats Plant in poly drums are returned directly to the poly drum for return to the Rocky Flats Plant.

### 4.1 Packing Samples in 1-Gallon Paint Cans

- 4.1.1 Place glass jars containing raw sample into individual, sealable plastic bags.
- 4.1.2 Place the jars into the bottom of the 1-gallon paint can, packing them as tightly as possible.
  - Note: Place jars from only one matrix into each can.
- 4.1.3 Pour vermiculite into the can to fill the spaces between the bags and to cover the bags.

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4.1.4 Place a second layer of jars in plastic bags into the can and cover with vermiculite. Repeat this until the can is full. Top off the can with vermiculite.

4.1.5 Close the paint can lid securely. Label the can with the matrix and a radioactive materials label.

#### 4.2 Packing Samples in 5-Gallon Plastic Pails

4.2.1 Place solidified materials from treatability studies into pails, filling each as full as possible.

4.2.2 Close the lid securely. Label the pail with the matrix and a radioactive materials sticker.

4.2.3 Place each pail into a garbage bag, twist and tape the bag to close. (This is done to keep the vermiculite from adhering to the pail.)

#### 4.3 Packing Steel Drums

4.3.1 Verify that the steel drum is labeled with a bar code (prepared by EG&G Rocky Flats) and is stenciled with "EG&G Rocky Flats, Rocky Flats Plant, Rocky Flats, Colorado" as shown in Figure 1.

4.3.1 Place two drum liners into the drum.

4.3.2 Spread 2 - 3 inches of vermiculite across the bottom of the drum.

4.3.3 Place a layer of 1-gallon paint cans or one 5-gallon plastic pail on the bottom of the drum.

Note: Do not mix matrices in the drum.

4.3.4 Pour 2 - 3 inches of vermiculite over this layer to fill the voids around the cans/pail.

4.3.5 Place a cardboard circle over the layer, and pour 2 - 3 inches of vermiculite over the circle.

4.3.6 Repeat 4.3.3 through 4.3.5 until the drum is full. Top off the drum with several inches of vermiculite.

4.3.7 Twist and tape, then fold and tape, the inner drum



liner. Repeat with the outer drum liner.

- 4.3.8 Place the drum lid with gasket over the top of the drum.
- 4.3.9 Place the lock ring around the top of the drum, positioned such that the locking bolt is at a 90° angle to the right of the drum seam. (See Figure 1.)
- 4.3.10 Torque the bolt to  $45 \pm 5$  ft. lbs.
- 4.3.11 Label the drum with the following, positioned as shown on Figure 1.
  - o Address label with laboratory name and address, and EG&G Rocky Flats and Rocky Flats plant address (Figure 2)
  - o "USA DOT-7A Type A Radioactive Material" label (Figure 3)
  - o "Radioactive LSA, N.O.S UN2912 (Pu-239)" label (Figure 4)
  - o Up-arrows label (Figure 5)
  - o Label showing contents (i.e., sample matrix) of drum (Figure 6)
- 4.3.12 Perform a smear survey and a radiation survey of each drum as described in LSG Procedure RS-8.
- 4.3.13 Weigh the drum and record the weight on the drum (Figure 6 label) and on the radiation survey record.

#### 4.4 Inspecting Steel Drums

Prior to shipment, a member of the LSG quality assurance department will inspect each steel drum for the following.

- o Proper identification on the drum
- o Proper labeling on the drum
- o Weight plainly marked on the drum

- o Each bung hole tightened
- o Locking ring at 90° angle to right of seam
- o Bolt torqued to 45 ± 5 ft. lbs.

The inspection is documented on a checklist (Figure 7) and the drums dispositioned as follows.

- o If the drum conforms to this procedure, custody seals are initialed and dated by the inspector, and placed over each bung hole and the locking bolt, such that they will break if a bung hole or the locking bolt are loosened.
- o If the drum does not conform to this procedure, the nonconforming condition is documented on the inspection checklist and brought to the attention of the RAM Coordinator for correction. Upon correction, the drum is re-inspected.

#### 4.5 Packing Poly Drums

- 4.5.1 Fill poly drums with liquid sample of one matrix.
- 4.5.2 Close and tighten the bung hole.
- 4.5.3 Label the drum with the drum identification number.

This will be assigned by LSG if EG&G has not provided a unique bar code identifier for the drum, as follows:

XXXXXX - YY

Where: XXXXXX is the month, date, year of the shipment and YY is a consecutively assigned number starting with 01.

- 4.5.4 Perform a smear survey and a radiation survey of each drum as described in LSG Procedure RS-8.
- 4.5.5 Weigh the drum and record the weight on the radiation survey record and drum label (Figure 6).

#### 4.6 Inspecting Poly Drums

Prior to shipment, a member of the LSG quality assurance department will inspect each poly drum for the following.

- o Proper identification on the drum
- o Proper labeling on the drum
- o Weight plainly marked on the drum
- o Each bung hole tightened

The inspection is documented on a checklist (Figure 7) and the drums dispositioned as follows.

- o If a drum conforms to this procedure, custody seals are initialed and dated by the inspector, and placed over each bung hole such that they will break if the bung hole is loosened.
- o If the drum does not conform to this procedure, the nonconforming condition is documented on the inspection checklist and brought to the attention of the RAM Coordinator for correction. Upon correction, the drum is re-inspected.

#### 4.7 Selecting a Carrier

The RAM Coordinator selects the carrier for each shipment. The carrier must meet the following criteria:

- o Certified interstate commerce commission motor common carrier.
- o EPA-licensed transporter authorized to transport radioactive waste, having an EPA identification number maintained on file by LSG.
- o Agree to provide sole use transportation to LSG for the shipment.

Note: Roadway Express, Inc. will not be used as a carrier based on their lack of cooperation regarding inquiries into a September 1992 shipment.

#### 4.8 Loading the Tractor Trailer

- 4.8.1 Prepare the following shipping documents to accompany the shipment. (See also section 5.)

- o LSG radioactive materials shipment record
- o Solar pond project packing slip
- o LSG chain of custody record

4.8.2 Place the drums onto wooden pallets according to the rules below. Band the drums together with steel bands.

- o All drums on a pallet must be of same material (i.e., all steel or all poly).
- o All drums on a pallet must all be of the same dimension (e.g., 55-gallon).
- o All drums on a pallet must be filled or all drums must be empty.

4.8.3 On the day of shipment, prior to loading the pallets onto the trailer bed, a member of the LSG quality assurance department will inspect each pallet in the shipment for the following.

- o Drums of same dimension, material, and all either filled or empty
- o Banding tight
- o Custody seals on each bung hole of each drum, and on locking bolt of each steel drum

4.8.4 When the truck arrives and following QAD inspection of the pallets, load pallets into the trailer bed. Distribute the load evenly and to prevent the pallets from shifting side-to-side under normal conditions.

When all of the pallets are loaded, brace the rear of the load so as to prevent the pallets from shifting front-to-back under normal conditions.

4.8.5 Document the following information on the carrier's bill of lading:

- o "To" and "From" addresses
- o Reference to "Instructions to Carrier" (Figure 11) and to additional instructions, if

any, on the chain of custody record.

- o General description of the shipment (same as description on the chain of custody record)
- o Proper DOT shipping name (same as chain of custody record)

4.8.6 Send the following documents to the EG&G Rocky Flats contact named as the recipient of the shipment:

- LSG radioactive material shipment record (Figure 8)
- LSG solar pond project packing slip (Figure 9)
- LSG chain of custody record (Figure 10)
- Bill of lading

#### 4.9 Inspecting the Trailer

4.9.1 After the pallets have been loaded onto the trailer bed, a member of the LSG quality assurance department will inspect the loading of the trailer and talk with the driver to determine the following.

- o Load braced
- o Trailer placarding correct
- o Driver has a copy of the following documents:
  - Bill of lading
  - LSG shipment packing list
  - LSG radioactive material shipment record
  - LSG chain of custody record, signed by LSG representative relinquishing custody and by the driver accepting custody.
- o Driver understands incident reporting and emergency contact procedures
- o Driver understands chain of custody procedures

4.9.2 When the shipment is determined to be conforming, the driver closes and locks the trailer door. The inspector then initials and dates, and applies a uniquely numbered custody seal such that it will be broken if the trailer door is opened. The inspector

records the custody seal number on the shipment chain of custody record.

## 5.0 RECORDS

### 5.1 LSG Radioactive Material Shipment Record

The LSG Radioactive Material Shipment Record (Figure 8) is completed by the LSG RAM Coordinator in accordance with LSG Procedure RS-8. The record includes the following.

- o "To" and "From" addresses
- o Date of shipment
- o Carrier
- o Special instructions (which may reference special instructions on the chain of custody record)
- o Description of shipment
- o Radionuclides, physical and chemical form, and activity in Ci, mCi, or uCi Pu-239.
- o Results of radiation and smear surveys of each drum
- o Labeling requirements
- o Certification that the shipment is properly classified, described, packaged, marked, and labeled, and is in proper condition for transportation according to applicable DOT regulations

### 5.2 LSG Solar Pond Project Packing Slip

The LSG Solar Pond Project Packing Slip (Figure 9) is completed by the LSG RAM Coordinator. The record includes the following.

- o "To" and "From" addresses
- o Gross weight of the shipment
- o Categories label (specify "none" if none apply)
- o Transportation index (specify "none" if none apply)

- o The following for each drum:
  - unique drum identification
  - composition of the drum (poly or steel)
  - description of the contents (i.e., the matrix)
  - EG&G IDC code
  - pallet number
- o Physical description of any other RAM being shipped (e.g., analytical equipment with internal contamination)
- o Signature of the RAM Coordinator

### 5.3 LSG Chain of Custody Record

The LSG Chain of Custody Record (Figure 10) is completed by the LSG RAM Coordinator. The record includes the following.

- o General description of the material in the shipment (e.g., unused sample, empty drums) and the total number of drums
- o Gross weight of the shipment
- o Proper DOT shipping name, hazard class, and identification number: Radioactive Material Low Specific Activity (LSA), N.O.S., UN2912 (Pu-239)
- o Space for relinquishing and accepting custody of the shipment
- o Custody seal number of the seal on the trailer door
- o Special instructions to the driver and recipient. Specific reference to and a copy of the Instructions to Carrier (Figure 11) is acceptable for the driver's instructions.

### 5.4 LSG Quality Assurance Inspection Checklist

The LSG Quality Assurance Inspection Checklist (Figure 7) is completed by the Quality Assurance inspector as the inspection is performed.

### 5.5 Bill of Lading

The carrier's Bill of Lading is expected to vary somewhat from

carrier to carrier. At a minimum, the RAM Coordinator must document the following on the Bill of Lading:

- o "To" and "From" addresses
- o Reference to "Instructions to Carrier" (Figure 11) and to additional instructions, if any, on the chain of custody record.
- o General description of the shipment (same as description on the chain of custody record)
- o Proper DOT shipping name (same as chain of custody record)

#### 5.6 Records Retained

LSG retains the following records from each shipment in accordance with the LSG radiation safety procedures.

- o Carrier's bill of lading
- o LSG chain of custody record
- o LSG radioactive material shipment record
- o LSG solar pond project packing list
- o Survey records from the radiation and smear surveys
- o Quality assurance inspection checklists

#### 6.0 APPARATUS

##### 6.1 Steel and poly drums: Provided by EG&G Rocky Flats, Inc.

Note: Steel drums must be labeled by EG&G with a unique bar code identifier and be stenciled with the company name. Return steel drums not labeled in this manner unused to EG&G Rocky Flats.

Steel drums must be supplied with a gasket and lid, locking ring, and appropriate nuts and bolts.

##### 6.2 Drum Liners: 8 mil or heavier. Obtained from EG&G Rocky Flats, Inc. or purchased commercially.



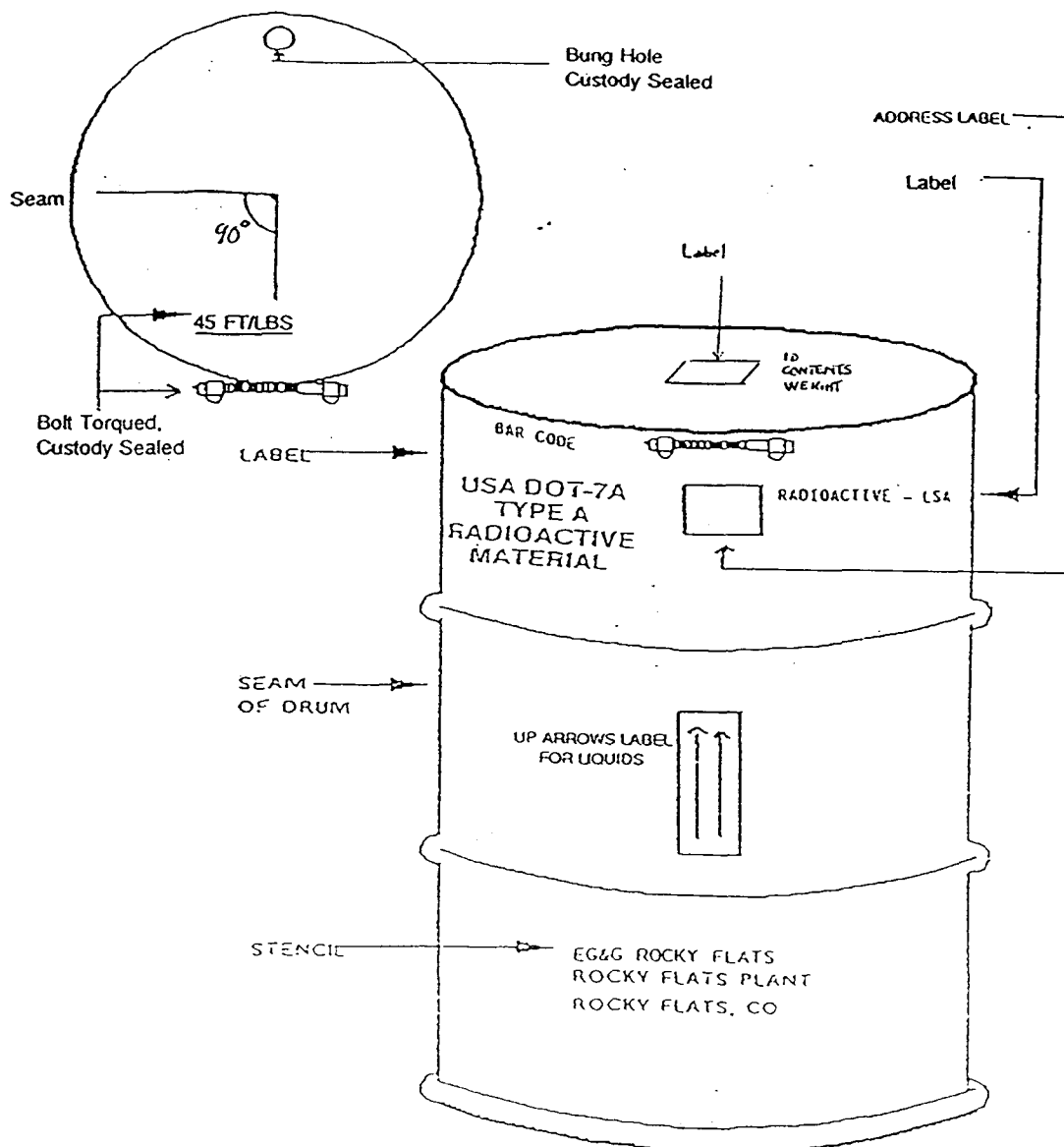
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- 6.3 Vermiculite.
- 6.4 Torque wrench: Capable of supplying at least 50 ft. lbs. torque.
- 6.5 Socket wrench.
- 6.6 Paint cans: 1-Gallon capacity, with lid.
- 6.7 Plastic pails: 5-Gallon capacity, with lid.
- 6.8 Plastic bags: Sealable.
- 6.9 Labels: See Figures 2 - 6.
- 6.10 Custody seals: With provision for initials and date. Custody seals used for the trailer door must be uniquely numbered.

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Figure 1

PONDCRETE/SALTCRETE SAMPLE DRUM



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Figure 2

HALLIBURTON NUS  
5350 CAMPBELLS RUN RD.  
PITTSBURGH, PA 15205

EG&G ROCKY FLATS, INC.  
ROCKY FLATS PLANT  
P.O. BOX 464  
GOLDEN, CO 80402-0464

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Figure 3

**USA DOT-7A  
TYPE A  
RADIOACTIVE  
MATERIAL**

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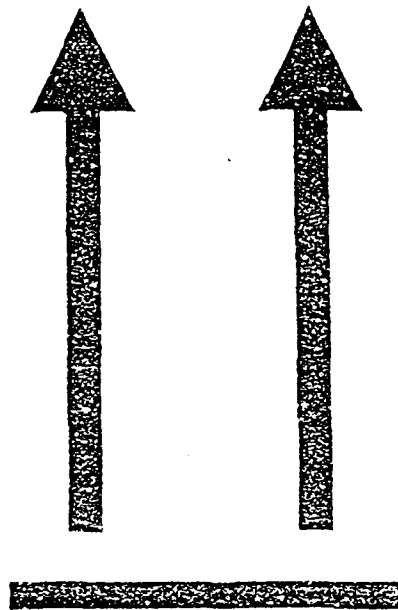
Figure 4

# **RADIOACTIVE MATERIAL**

## **LSA, N.O.S., UN2912 (Pu-239)**

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Figure 5



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Figure 6

ID # \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_ LBS.

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Figure 7

SOLAR POND PROJECT  
QA INSPECTION OF PALLETS AND TRAILER LOADING

Reference: LSG Procedure AP-017, Section 4.0

Date: \_\_\_\_\_  
Carrier: \_\_\_\_\_  
Trailer Number: \_\_\_\_\_

Y/N

Drums segregated on pallets by construction (steel/poly): \_\_\_\_\_  
Drums segregated on pallets by size: \_\_\_\_\_  
Drums segregated on pallets by content (full/empty): \_\_\_\_\_

Load braced: \_\_\_\_\_  
Trailer placarded I.A.W. 49 CFR 172.504, 172.556: \_\_\_\_\_

Driver has bill of lading: \_\_\_\_\_  
Driver has packing list: \_\_\_\_\_  
Driver has radioactive material shipment record: \_\_\_\_\_  
Driver has signed chain of custody record: \_\_\_\_\_

Driver understands emergency procedures (incident reporting): \_\_\_\_\_  
Driver understands sole use status: \_\_\_\_\_  
Driver understands chain of custody procedures: \_\_\_\_\_

Draw a diagram of how the trailer was loaded on the back of this form.

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Inspected By: \_\_\_\_\_  
Date: \_\_\_\_\_

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SOLAR POND PROJECT  
QA INSPECTION OF DRUMS PREPARED FOR SHIPMENT

Reference: LSG Procedure AP-017, Section 4.0

Drum ID	Labelled Fully	Weight Marked	Bung Hole Tight	Locking Ring at 90°	Torque at 45±5 ftlbs	Custody Seal Applied

C o m m e n c e s

Inspected By: \_\_\_\_\_  
Date: \_\_\_\_\_

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Figure 8

RADIOACTIVE MATERIAL SHIPMENT RECORD

TO: \_\_\_\_\_ DATE: \_\_\_\_\_ FROM: \_\_\_\_\_  
CARRIER: \_\_\_\_\_  
COMMENTS: \_\_\_\_\_  
ATTN: \_\_\_\_\_ SHIPPER: \_\_\_\_\_  
CONSIGNEE LICENSE NO. \_\_\_\_\_ TELEPHONE NO. \_\_\_\_\_

SHIPMENT DESCRIPTION

Reason for Shipment:

Physical Form: \_\_\_\_\_ Solid \_\_\_\_\_ Liquid \_\_\_\_\_ Gas  
Form: \_\_\_\_\_ Normal \_\_\_\_\_ Special \_\_\_\_\_ Instrument/Article  
Chemical Form: \_\_\_\_\_  
Shipment Category: \_\_\_\_\_ Nonradioactive (less than 0.002 microcuries/gram) \_\_\_\_\_ Limited Quantity, Instrument or Article  
\_\_\_\_\_ LSA \_\_\_\_\_ Type A

Radioactive Material Description:

Package Description: \_\_\_\_\_ Strong, Tight \_\_\_\_\_ Essentially Type A \_\_\_\_\_ Type A  
Package Gross Weight: \_\_\_\_\_ Package Gross Volume: \_\_\_\_\_

CONTENTS/MONITORING DATA

Radionuclides	Specific Activity	Total Activity ____ $\mu$ Ci ____ mCi ____ Ci	Package Radiation Level		Removable Contamination		Date of Survey	Surveyor's Name and Initials
			Surface	1 Meter	Alpha	Beta		

LABELLING

\_\_\_\_\_ None required, limited quantities, instruments or articles  
\_\_\_\_\_ Radioactive White I  
\_\_\_\_\_ Radioactive Yellow II  
\_\_\_\_\_ Radioactive Yellow III

CERTIFICATION

\_\_\_\_\_ This is to certify that the above named materials are properly classified, packaged, marked, and labeled and are in proper condition for transportation according to the applicable regulations of the Department of Transportation.  
\_\_\_\_\_ This shipment is within the limitations prescribed for passenger/cargo-only aircraft (delete non-applicable).  
\_\_\_\_\_ This package conforms to the conditions and limitations specified in:  
\_\_\_\_\_ 49 CFR 173.421 for excepted radioactive material, limited quantity, n.o.s. UN2910.  
\_\_\_\_\_ 49 CFR 173.422 for excepted radioactive material, instruments and articles, UN2911.  
\_\_\_\_\_ 49 CFR 173.424 for excepted radioactive material, articles manufactured from natural or depleted uranium or natural thorium, UN2909.

SIGNED BY: \_\_\_\_\_  
(for shipper)

DATE: \_\_\_\_\_

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Figure 9

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HALLIBURTON NUS ENVIRONMENTAL CORPORATION  
SOLAR POND PROJECT PACKING LIST

Ship To:

EG&G Rocky Flats, Inc.  
Rocky Flats Plant  
Rocky Flats, Colorado 80403  
Attention: \_\_\_\_\_

From:

HALLIBURTON NUS Environmental Corporation  
Laboratory Services Group  
5350 Campbells Run Road  
Pittsburgh, Pennsylvania 15205  
Contact: \_\_\_\_\_  
Phone Number: \_\_\_\_\_

Gross Shipment Weight: \_\_\_\_\_

Categories Label: \_\_\_\_\_

Transportation Index: \_\_\_\_\_

Pallet #	Drum #	Steel/ Poly	Contents	IDC Code
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____
_____	_____	_____	_____	_____

Other Material: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Released for Shipment by: \_\_\_\_\_

Title: \_\_\_\_\_

Date: \_\_\_\_\_

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Figure 10

### CHAIN-OF-CUSTODY RECORD

Date \_\_\_\_\_

COC No. \_\_\_\_\_

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Carrier (Company Name)			Consignee Name and Address		
Shipper					
Address					
City	State	Zip Code	Destination City	State	Zip Code
Origin City (if different)	State	Zip Code	Phone Number:		

No. Shipping Units	Kind of Packaging	Weight (kg)	Description of Articles, Special Marks, and Exemptions	DOT Shipping Name

Chain-of-Custody for Shipment			
Relinquished by: (signature)	Date/Time:	Received by: (signature)	Date/Time:
Relinquished by: (signature)	Date/Time:	Received by: (signature)	Date/Time:
Relinquished by: (signature)	Date/Time:	Received by: (signature)	Date/Time:

Truck Door Custody Seal					
Seal Number	Placed on Door By	Date/Time	Broken By	Date/Time	Circumstances Under Which Seal Broken

Special Instructions	
To the Driver:	
To the Consignee:	<p>(a) Accept custody of the shipment by signing and dating the Chain-of-Custody for Shipment section of this form.</p> <p>(b) Document breaking the truck door custody seal by signing and dating the Truck Door Custody Seal section of this form. Note any irregularity with the custody seal.</p> <p>(c) Note condition of the shipment upon arrival below:</p> <p>(d) Return the indicated copy of this signed form to the address as follows:</p> <p>HALLIBURTON NUS Environmental Corporation 5350 Campbell's Run Road Pittsburgh, PA 15205 Attn:</p>

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## Figure 11

### INSTRUCTIONS TO CARRIER FOR SHIPMENT OF MATERIALS TO THE ROCKY FLATS PLANT ROCKY FLATS, COLORADO

Prepared by HALLIBURTON NUS Environmental Corporation -- Nov. 1992

1. Delivery to the Rocky Flats Plant
  - o Deliver this shipment to the Rocky Flats Plant Monday through Friday during normal business hours only.
  - o Notify the EG&G contact, identified as the consignee on the chain of custody record, 3 hours prior to arrival at the plant.
2. Reporting Incidents

Report any of the following incidents involving this shipment to the Department of Transportation (DOT) as required under the provisions of 49 CFR 171.15.

  - o A person is killed.
  - o A person receives injury requiring hospitalization.
  - o Estimated carrier or other property damage exceeds \$50,000.
  - o Fire, breakage, spillage, or suspected radioactive contamination occurs involving the radioactive materials in this shipment.

Notify the DOT at the earliest practicable moment at:

1-800-424-8802

Provide the following information:

- o Your name.
- o Name and address of the carrier.
- o Phone number where you can be reached.
- o Date, time, and location of the incident.
- o The extent of injuries, if any.
- o Classification, name, and quantity of hazardous material involved.
- o Type of incident and nature of hazardous material involvement, and whether a continuing danger to life exists at the scene.

Report the same information to HALLIBURTON NUS Lab Services Group at:

1-800-228-6870 (business hours)  
or  
1-800-255-3924 (24-hour emergency number)

In addition, report any incidents not usually encountered, such as inspection of the shipment by the DOT or equivalent state or local agency, to HALLIBURTON NUS Lab Services Group.

### 3. Sole Use Status

This vehicle is assigned for sole use of HALLIBURTON NUS Laboratory Services Group under provisions of 49 CFR 173.425(b). The load is not to be adjusted, transferred to another vehicle, tampered with, or broken.

If, for any reason, the custody seal on the trailer is broken prior to delivery to the consignee, the occurrence must be documented on the chain of custody record, to include an explanation of the occurrence, signature, date, and time. A new seal must be attached to the trailer door, and the new seal number documented on the chain of custody record.